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DESIGN AND DEVELOPMENT OF A X-RAY CAMERA FOR CONTINUOUS POWDER PHOTOGRAPHS DURING PROGRAMMED TEMPERATURE CHANGES

by

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ABSTRACT

X-ray Analysis, using the Debye-Scherrer method, has been a laboratory tool for many years in studying the behavior of materials. The importance of performing investigations at high temperatures and changing environments led to the design and development of a X-ray Camera that extends the capabilities of basic powder pattern technology. A camera was designed to take a continuous sequence of x-ray powder photographs on a single strip of film while specimen temperature was changed. The design and development of this apparatus and its application in phase transformation studies is described and illustrated. The powder pattern photographs obtained with this apparatus provide desirable features and comparison presentation that can be applied to other environmental investigations.

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1. Introduction.

In man's quest to extend his frontiers from the comfortable confines on the surface of Mother Earth to the unknowns of outer space and forbidding depths of the sea, there have been increased demands on materials to withstand extreme ranges of temperatures, pressures, and other adverse conditions. Our success or failure in the future, as in the past, depends on man's fuller understanding and imaginative use of the materials available. There are many tools, both microscopic and macroscopic, which enable man to probe deeply into the mechanical and physical behavior of materials.

One of the tools of the twentieth century is X-ray analysis. From the outset of the discovery of diffraction of X-rays by crystals and consequent recording on a film, it was recognized that here was a tool to be adapted for detailed study of a large group of materials, including metals and their alloys, which are characterized by a crystalline state. X-ray analysis reveals the nature of the metal showing how the atoms are placed relatively to each other. One method of X-ray analysis is known as the Debye-Scherrer or powder method and is especially attractive to the metallurgist since it does not require single crystals of metals which are difficult to obtain. Fundamentally, the powder method provides a way of investigating the crystallography of the crystal powder. The powder method provides the following for the investigator with varying de-

grees of accuracy as compared to other methods.[3,8,9,12,13]

(1) Precise lattice constants

(2) Phase identification

(3) Solid-solution studies

(4) Stress measurements

It was recognized, shortly after the powder method was discovered, that it would be very desirable to observe the possible changes in the structure of a substance after the environment was altered. Temperature effects on material behavior seemed the most significant of the environmental factors, and therefore more research has been done in this area than in all the others combined. Debye cameras, adapted for high temperature work, vary from laboratory to laboratory since the criteria are subject to the demands of the user. [15,18,23] One demand for a high temperature camera is the determination of phase transformation temperatures, which are used in phase diagram construction.[2,6,8,14]

In the past the data for constructing the phase diagram for any system of metals has been obtained by some secondary method or methods, such as heating or cooling curves, dilatometer measurements, specific heat curves, resistivity curves. Occasionally, X-ray diffraction powder patterns have been used, but only on specimens which have been quenched from the temperature for which the data are desired. The unreliability of the above methods led to a primary and reliable method of obtaining phase diagram data by means of a controlled-temperature X-ray diffraction technique.[2] The method

consists of heating the specimen to the desired temperature in a specially designed small furnace which is contained in the X-ray powder camera; a diffraction pattern of the material is then obtained while the specimen is held at this elevated temperature. For a specimen of specified composition, a series of photographs are taken at various temperatures. Since each phase has its own unique and distinctive pattern, a phase change or a change from one phase to two phases or the reverse may be detected easily.

The primary method described above has, in common with all known previous methods, one feature which is a disadvantage - it is time consuming. The following procedure for making a temperature study of a specimen of specific composition illustrates the tasks and time involved. The specimen is heated in the camera furnace to the desired temperature and when equilibrium conditions prevail, a diffraction pattern is obtained; the imput to the furnace is then increased to raise the specimen temperature to that next desired; the previously taken film is developed; an interval of time is allowed to elapse for equilibrium conditions to obtain before the next pattern is photographed; this process is then repeated for as many temperatures as are necessary for the required data to be determined. A single run for a specimen of specified composition may take several days of continuous work for the investigator.

This primary or single exposure method was further refined by using a controlled-temperature furnace adapted for

use on a Weissenberg X-ray Goniometer. This apparatus allows a series of diffraction patterns to be recorded on a single film, obviating the necessity of individually developing each pattern obtained in a run. Figure 21B shows the resulting series of powder patterns using this apparatus.

N. W. Buerger, who developed the controlled temperature X-ray diffraction techniques as described in the previous paragraphs, has further suggested a camera which could take a continuous series of exposures on a single sheet of photographic film, while the specimen is subjected to programmed temperature changes. This concept would permit all the X-ray diffraction patterns of the subject material to be recorded on one film strip and facilitate comparison of the patterns. Phase transformation temperature investigation as indicated above is one area where an apparatus based on this concept would be advantageous. The objectives of this research are the development of such an apparatus and the determination of its feasibility and application in phase transformation studies.

2. Literature Review.

Before any apparatus was designed to be used for examining the relative merits of the concepts proposed, a general study of high temperature X-ray equipment and procedures was conducted. The references contained in the Bibliography cover the literature on high temperature X-ray analysis in general from 1940 to the present. Although many different camera-furnace combinations and high temperature technology are described in these references, it was noted that the concepts and camera proposed for this research had not received previous attention with the exception of the phase diagram determination studies discussed in the introduction[2]. However, the author toward the end of his experimental work found reference to a camera which has similarities in objectives to the one described in this report[14]. The camera cited is commercially available from the Central Research Laboratories, Inc., Red Wing, Minnesota, and is capable of operating at 1000°C. It is designed with the specimen rotating on a horizontal axis with a movable carrier which allows five separate 7-mm wide patterns at selected temperatures to be taken on a single strip of film. Differences between the Central Research camera and the one designed and used in this research are apparent in the body of this report.

3. High Temperature X-ray Technology.

The interest in high temperature X-ray cameras dates back to 1921. Discarded from the outset was the possibility of enclosing the entire camera in a thermostatically controlled chamber since the film deteriorates rapidly above 70°C. Development of a more stable film was dropped in favor of developing a device to heat the specimen separately, while keeping the film cool by means of a radiation shield or cooling coils around the heater. Furnace designs are intricate and varied and can be placed into four heating classes [2,4,9,15,18,23,24].

(1) Passage of electric current through wire specimens.

(2) Induction heating through the specimen, or if that is not possible by induction heating of a specially prepared specimen container.

(3) Focused high intensity light rays - a type of solar furnace.

(4) Electric resistance heaters.

Each heating class has its own merits and all have difficulties in temperature measurement, which increase in magnitude as higher temperatures are required [12].

Of the various methods to measure specimen temperatures the principal one is by means of thermocouples [11,17]. Thermocouple procedures followed by researchers in the past have been quite varied in an effort to overcome the problems that are imposed by the size, shape, and location of the specimen being measured. These problems contribute to

errors in temperature measurement, and the majority of reported experimental work avoids detailed discussion on error analysis [23]. Another method of temperature measurement uses an internal standard [20]. It is preferable to thermocouples at higher temperatures where a greater degree of accuracy is desired. In this method sample temperatures are determined by comparing expansion measurements from powder patterns of a material, such as platinum, intimately mixed with the sample, to a standard expansion curve of the added material. The expense of the internal standard and the complexities in specimen preparation limit its use. An additional method of determining specimen temperature, with a high degree of accuracy, is to calibrate the heating unit using various polymorphous substances of known transformatemperatures [2,4,10]. The polymorphic specimen is mounted in the camera and a series of powder photographs are taken at known energy imput to the heating element. The transformations are noted in the photographs and a temperature-energy calibration chart may be plotted. This reduces the effect of temperature gradient of the furnace to a minimum, since one is concerned only with the temperature of the portion of specimen giving rise to the diffraction pattern.

Another difficulty encountered in furnace design concerns temperature gradients across the specimen. Temperature gradients contribute to line broadening of the X-rays being diffracted, and this is most undesirable if the powder patterns are to be used in the determination of exact

lattice parameters. However, temperature gradients are more of a problem in diffractometer specimens, than in powder specimens, and when using metallurgical powder specimens, temperature gradients can be considered negligible due to their high thermal conductivity.

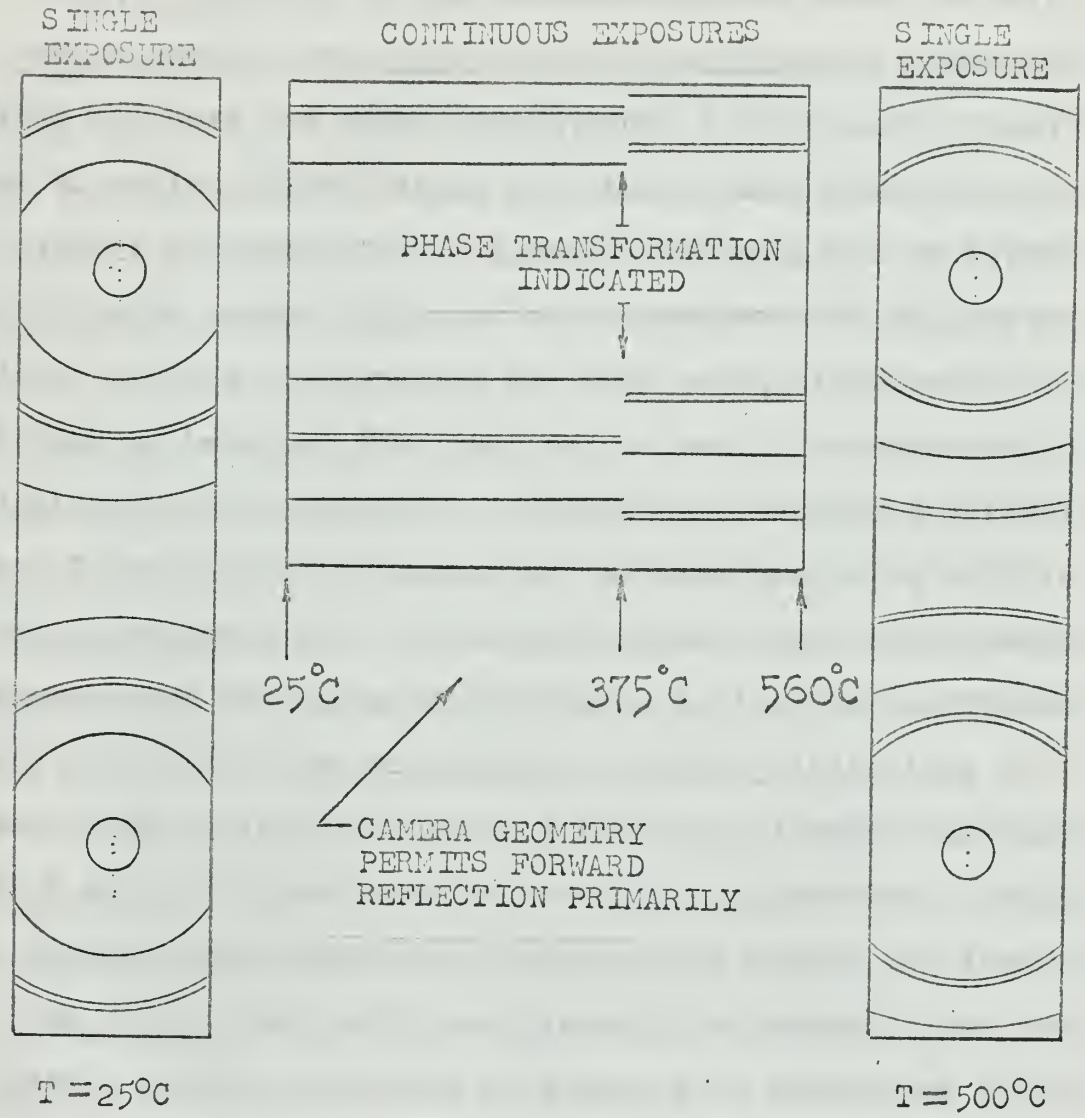
4. Proposed Concept and Applications.

The Debye-Scherrer or powder method employs monochromatic radiation falling on a powdered specimen or fine grained polycrystalline specimen, and produces a single diffraction pattern on a strip of photographic film. This research modifies the standard camera by taking a continuous sequence of powder patterns on one sheet of photographic film. This entails moving the film across the path of the diffracted X-rays in much the same manner that the Weissenberg camera moves horizontally across the diffracted X-ray path. However, the concepts of this research require the horizontal movement to attain a series of powder patterns for a different reason than that employed in the Weissenberg method. It will be recalled that in the introduction section of this paper, components of the Weissenberg were used for an apparatus to obtain a series of powder photographs on one film strip. The components used did not include the Weissenberg's mechanism for continuous horizontal travel. For the proposed camera, as compared to the Weissenberg camera, a much slower horizontal advancement is required since translation speed is determined by exposure time of the sample being used. During the horizontal movement of the film the temperature of the specimen will be changed at a slow rate endeavoring to observe and compare diffraction pattern changes on a single strip of film. This entails heating the specimen at a predetermined rate correlated with the exposure period.

By means of a hypothetical example, the concept of this research will be repeated by the following illustration. From a powder or thin solid specimen mounted in a furnace, a X-ray powder pattern will be focused on a small section of a long photographic film strip. The film strip moves across the focused X-ray diffraction area at a speed coincident with the necessary exposure time for the specimen and radiation used, and results in a continuous series of powder patterns recorded on the film. At the same time the film is moving, the specimen will be heated at a predetermined rate. If the temperature increase is slow enough, a possible slope to the lines of the powder pattern may result. This slope should be in direct proportion to the rate of thermal expansion of the sample material. If the sample is polymorphic, a definite change in pattern would be evidence of a phase transformation.

Figure 1 shows the author's conceptual sketch depicting a standard powder pattern at room temperature, a film strip with a continuous series of powder patterns, and a powder pattern at an elevated temperature, which illustrates pictorially the anticipated results of the example presented in the last paragraph.

In applications to specimens of unknown composition the following procedure is proposed for phase transformation investigation. The apparatus proposed could be used for a survey stage in specimen investigation, by scanning a predetermined temperature range and having the results on one



Conceptual sketch of X-ray Diffraction Powder Patterns obtained when using the proposed Continuous Exposure Camera and Single Exposure Camera at the temperatures indicated.

Fig. 1

film strip. This survey information provides the investigator with small temperature ranges of interest. These temperature ranges could be examined more closely by decreasing the rate of temperature change per exposure during a second camera pass, and if a phase transformation is observed by a change in pattern its transformation temperature could then be determined to a high degree of accuracy. It is not anticipated that the pattern will yield a sufficient number of lines of the sharpness required to enable lattice parameter measurements. Therefore the investigator at this point must remove the specimen and use one of the conventional high temperature cameras for portions of the investigation dealing with specific parameter measurements. This application does not necessarily cut down on the time that was formerly required in the investigation described in the introduction, but what is more important, the dark room time, camera manipulation, and temperature control is cut to a minimum and thus relieves the investigator of these burdensome time consuming endeavors. Other advantages are that one film strip provides the entire record for the investigation, and two or more related specimens can be interchanged and recorded on a single film strip for ready comparison. Further the basic concept of moving the film across the X-ray path and recording continuous powder patterns could be employed for other environmental variables affecting the specimen. Temperature was selected for this research for reasons specified in the introduction.

5. Description and Operation of Apparatus.

Figures 2 and 3 show the camera assembly with the major components labeled. Before designing the components a cardboard model was constructed to show the relative size of the apparatus, and give an overall idea of how the parts would mate together. The working drawings and parts list are given in the Appendix. The material used for the entire assembly is brass and is of a heavier construction than is actually necessary. Brass was used for ease in machining and was available from the U. S. Naval Postgraduate School Machine Shop in 3/4 inch plates of the length and width dimensions required. The camera was designed using 3/4 of an inch as a basic thickness dimension, wherever practical to save time on machining operations.

Figure 4 shows the camera structure, without the film cassette and main support post with attachments, in alignment with the North American Philips X-Ray unit. The collimator holder is attached to the sliding support base, and can be adjusted to provide alignment to the center line axis of the specimen. The sliding support base serves to move the entire upper assembly away from the X-ray exit port and provides an additional means for perpendicular positioning of the collimator to the specimen. The three screw legs permit the entire assembly to be leveled on the X-ray machine and allow perpendicular alignment of the collimator to the X-ray exit port, which on the Philips unit is positioned at an angle less than 90°C from the horizontal.

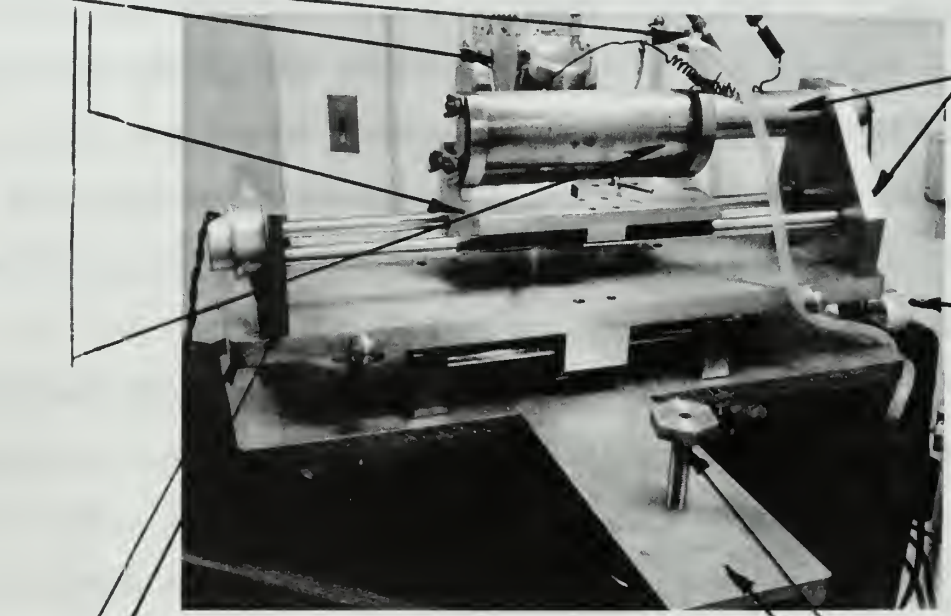
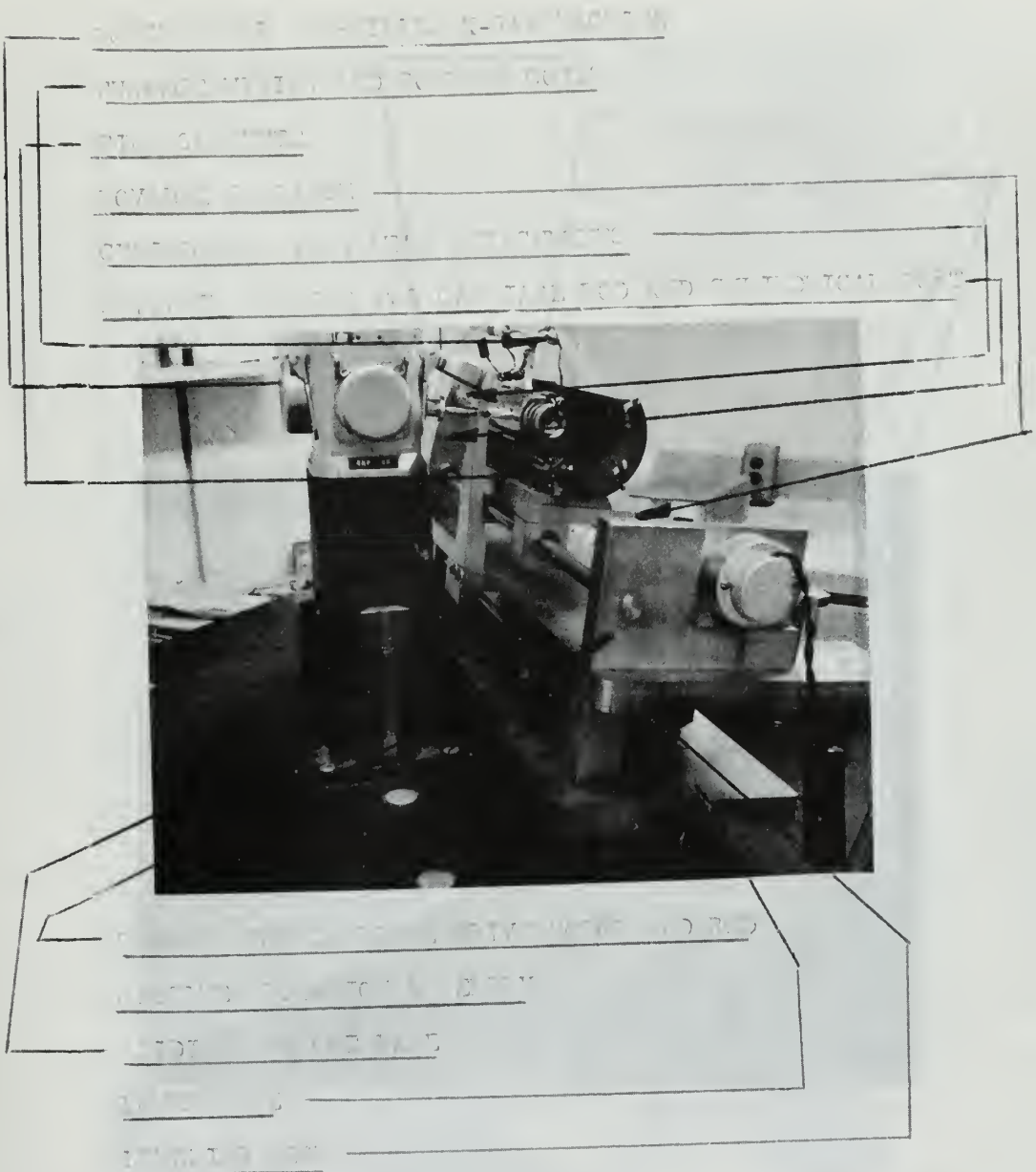


FIG. 2



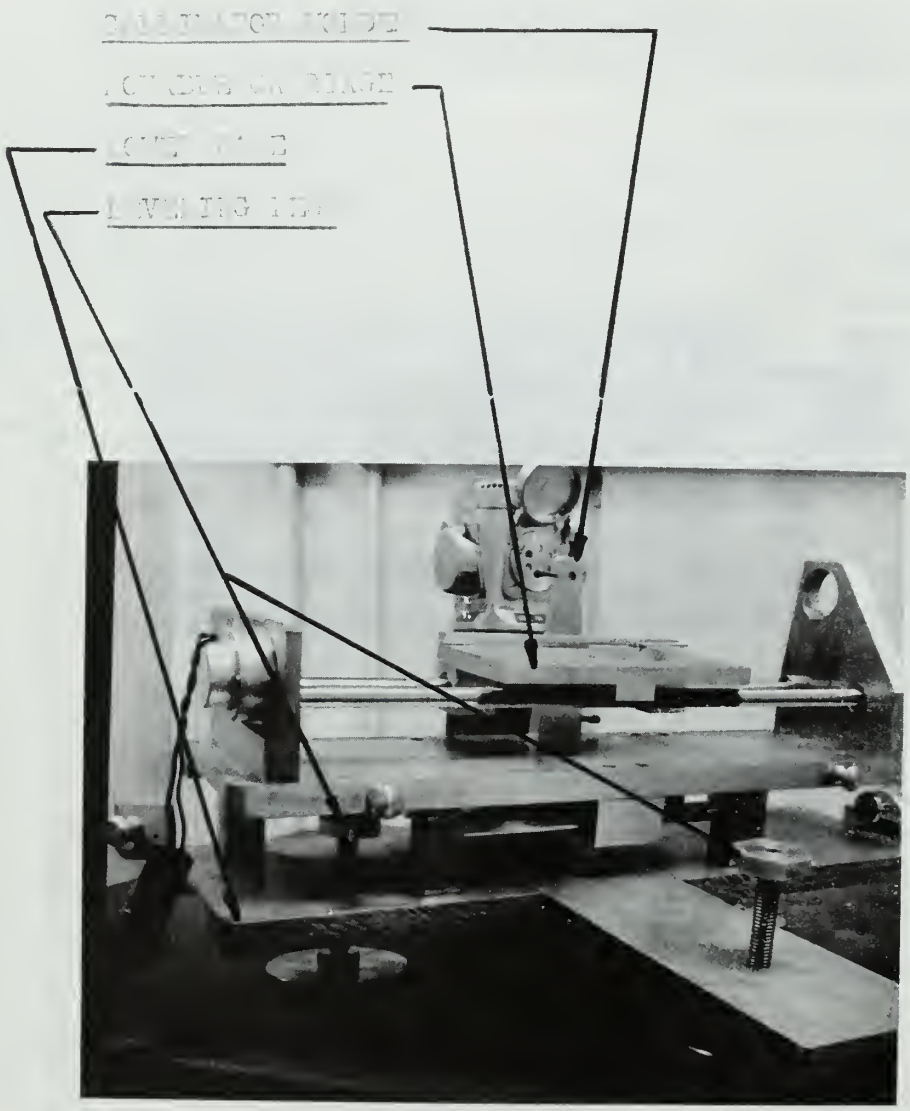


Fig. 4

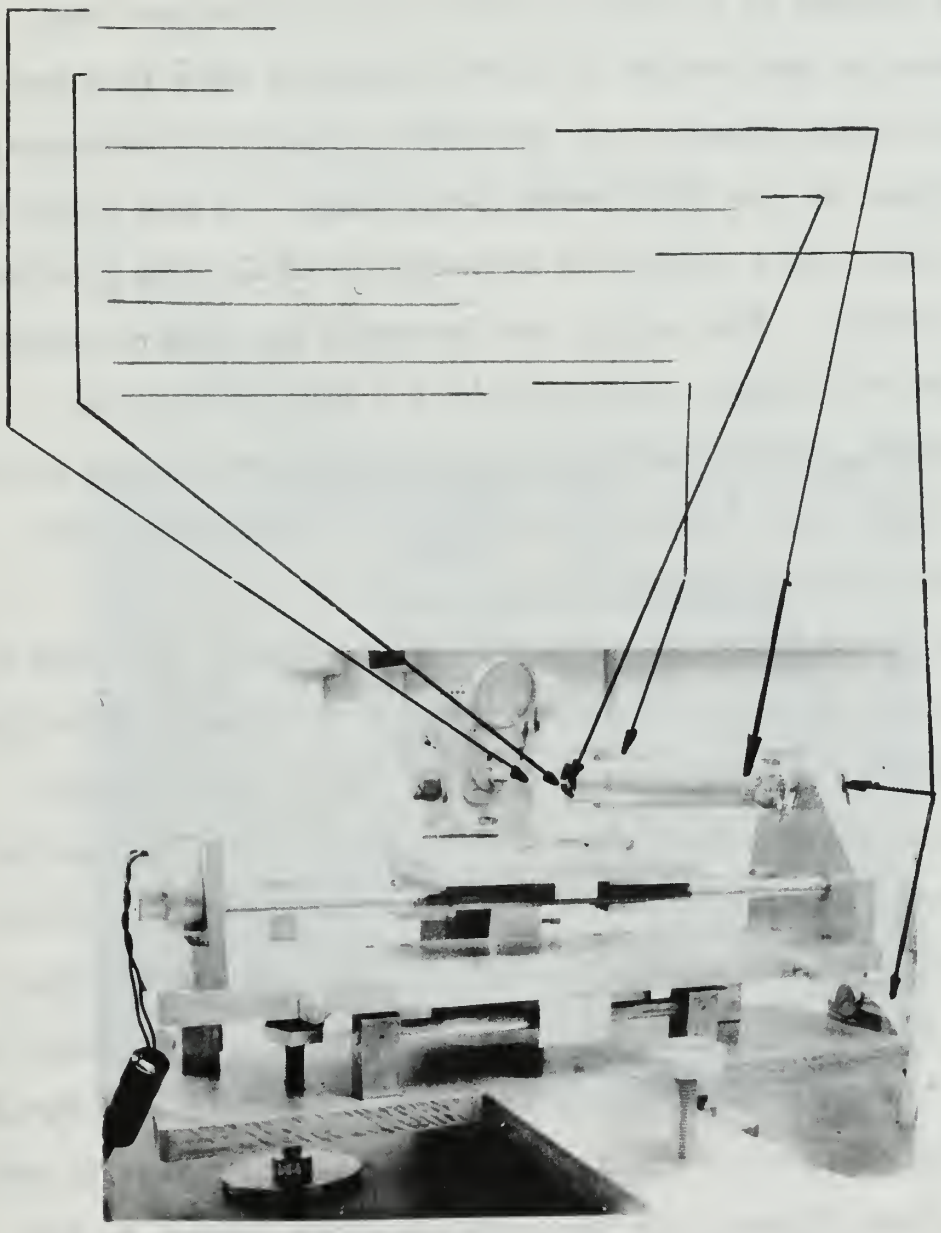


FIG.

Figure 5 shows the cylindrical post used to support the specimen holder, the furnace, and X-ray pattern focusing attachment. The specimen holder consists of an eccentric cam device attached to a 1/8 inch drill rod 11½ inches long. The eccentric cam device is of the standard type employed in other powder cameras and provides a means for centering the specimen to the collimated X-ray beam. At the right end of the drill rod a pulley is attached by which the specimen can be rotated. The pulley and accompanying pulley cable (oversized "O" Ring) are driven by a 1 RPM synchronous timing motor, manufactured by Bristol and distributed by the Minarik Elec. Co., Los Angeles, Calif., attached to the lower base of the apparatus.

The furnace, which surrounds the specimen, is shown in Figure 6 in its operating position. It is a resistance type heater with a heating capacity up to 800°C, and is constructed to permit rapid temperature change. It is part of a matching powder camera, and is described in detail by its designers.[4] This camer-furnace combination was manufactured by Otto von der Hyde and has been used for a number of years by the Material Science Department of the U. S. Naval Postgraduate School. A detailed look at the furnace is not possible from Figure 6 as the water cooling jacket is the most distinquishing feature shown and conceals the inner components. The major components not shown are the heating element, radiation barriers, and mountings. The heating element is a coil of chromel wire on the inside of a slotted

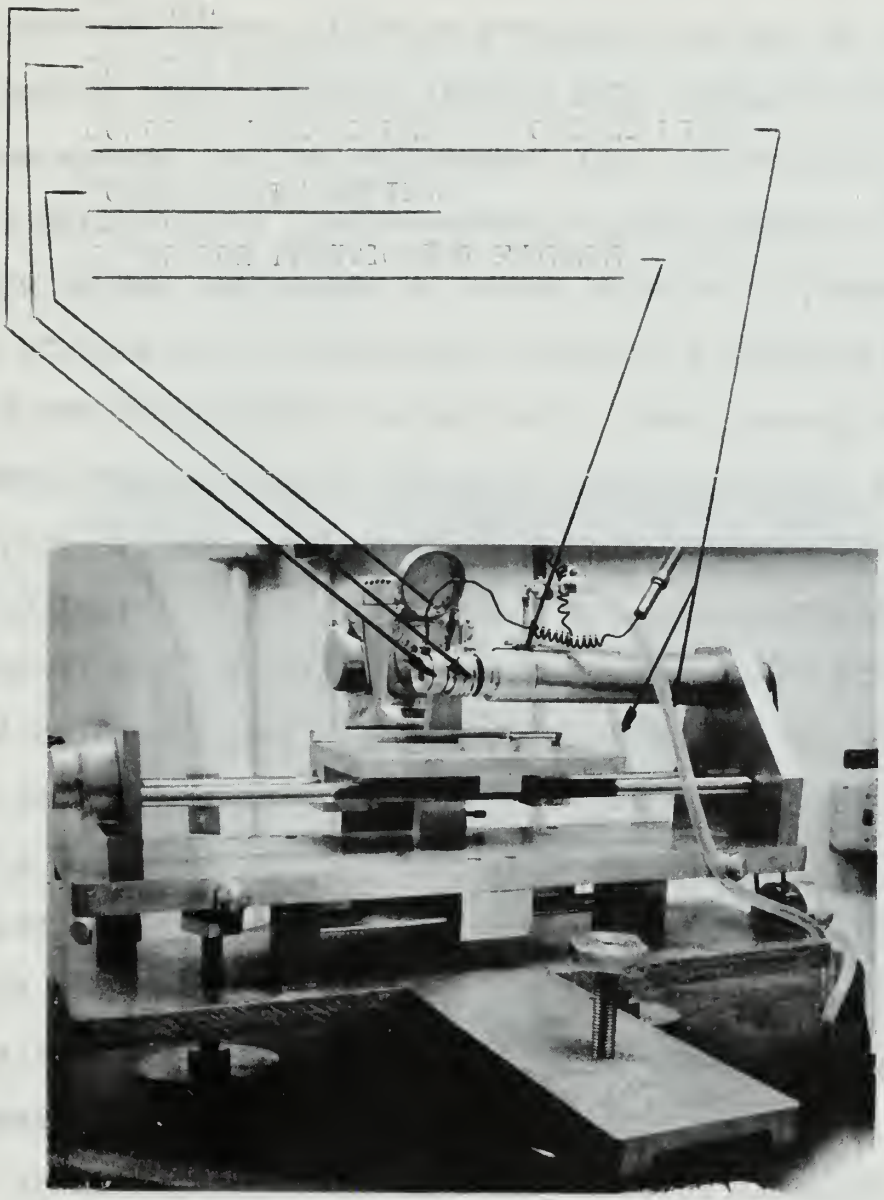


Fig. 9

porcelain tube which surrounds the specimen coaxially. The temperature is maintained by radiation barriers of aluminum cylinders of low heat capacity providing rapid specimen temperature changes. The visible cooling jacket protects the film against the high temperature of the furnace and provides a ground level of temperature. By providing a ground level, a definite amount of electrical energy will raise the specimen a definite temperature interval, ΔT, above this ground level. The source of energy for the furnace is a voltage/current regulated direct current power supply. It is manufactured by Kepco Inc., Mod ch 18-3, and its operation will be described later in this report.[21]

Figure 7 shows the X-ray diffraction pattern focusing attachment positioned over the furnace and maintained in proper position by a guide arm on the cylindrical post. The focus attachment, utilizing either a 1/8 inch or 1/16 inch wide slit, serves to confine the diffraction pattern to a narrow vertical strip on the film. A 5/16 inch hole located on the slit cylindrical center line and opposite the slit provides a means for the collimated X-ray beam to enter. Directly opposite the collimator entry hole is a movable exit collimator with a lead shield used as a beam catcher. The slit is 180° in circumferential length and located to be compatible to the film cassette. For a better understanding of the focusing attachment and location of the slit and the collimator entry and exit holes, the reader is referred to the Appendix where a working drawing can be

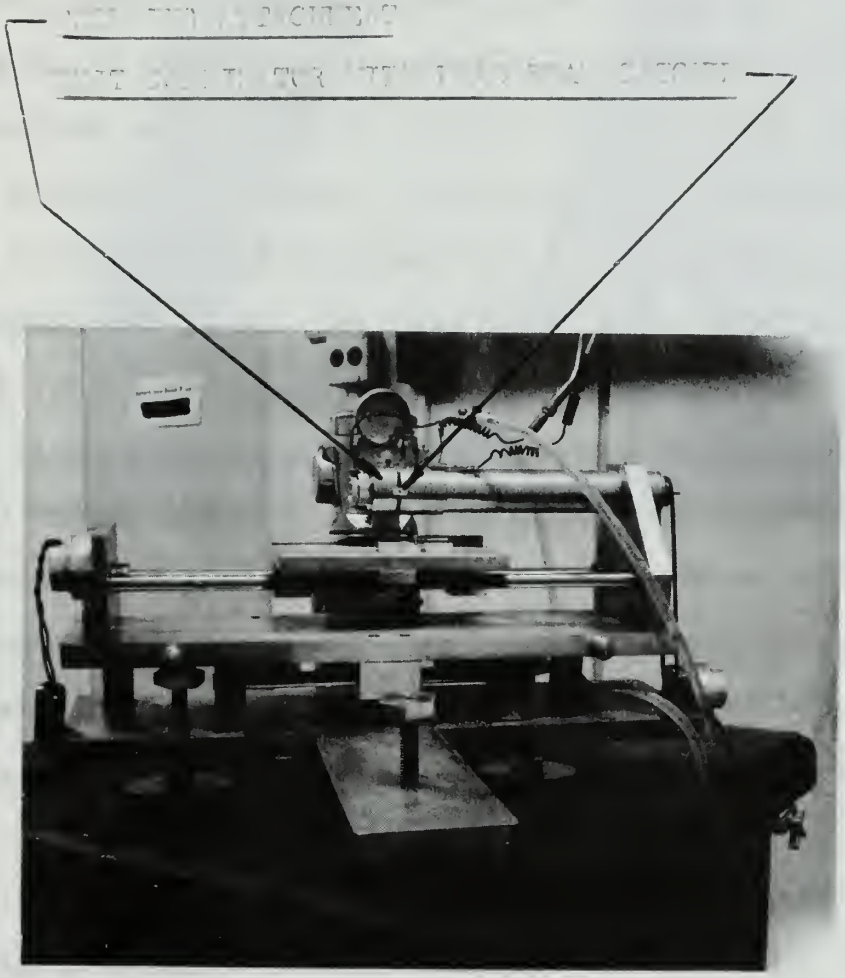


Fig. 7

found.

Figure 8 shows the carriage with film cassette in its extreme left hand or starting position. The carriage carries the film cassette to the right as viewed in the figure at very slow speeds across the path of the focused diffraction pattern. This carriage is driven by an accurately machined threaded rod and guided in its horizontal travel by means of a second rod located in precision slide bearings attached to the carriage. The drive rod is 1/2 inch in diameter with 56 threads per inch. The number 56 was selected for the following two reasons. First, a fine thread is required for smooth advance. Secondly, it is the preprogrammed thread cutting speed, of the "Lebold" Lathe used in its manufacture, that comes closest to 60 threads per inch, the desired number for quick calculation purposes.

The following rough calculation was used to determine the motor speeds required for the experimental work anticipated.

(1) Assume 60 threads per inch.

(2) This implies that for a motor speed of 1 RPM the carriage would advance 1 inch in 1 hour.

(3) Knowing that the slit size on the diffraction focusing attachment is either 1/8 or 1/16 inch implies eight or sixteen exposures side by side in one inch. Consequently the exposure times are 7.5 minutes and 3.75 minutes respectively for a 1 RPM motor.

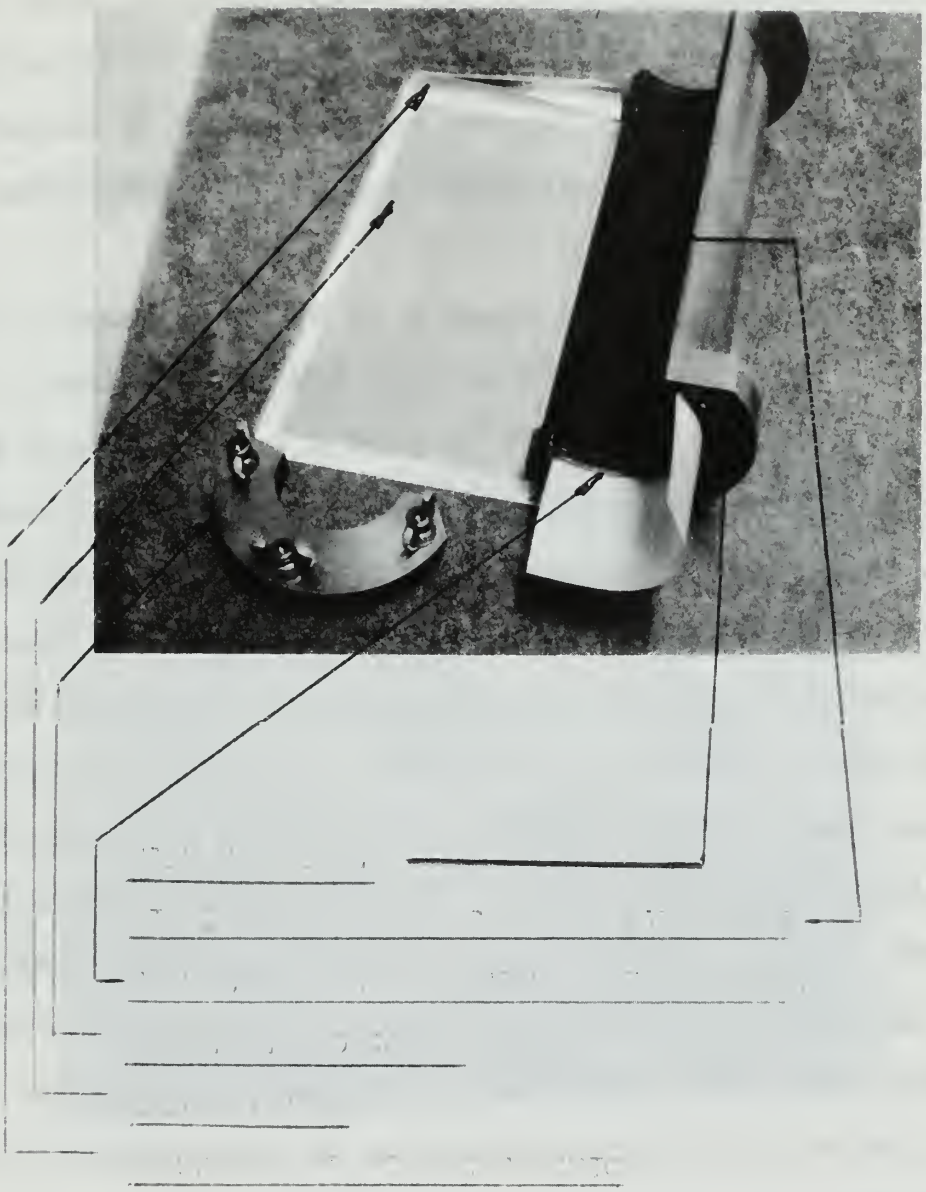


Fig. 8

(4) The anticipated exposure times range from 30 minutes to 2 hours, and are based on experimental work using the single exposure high temperature cameras. Also increases in these experimental times are necessary due to cassette absorption, capillary reflection, and higher temperatures.

(5) Therefore from steps 1 to 4, motor speeds in the range of 1/4 RPM to 1/32 RPM are required.

It is rather difficult to find a variable speed motor at the slow speeds required, and for that reason a number of small fractional speed synchronous timing motors of the type described for specimen rotation were selected to cover the above range of speeds. The motor drive is coupled to the drive rod by a close tolerance fit and locking set screw, and can easily be changed when a different carriage speed is required. The carriage lifts from the threaded portion of the rod and can slide to another desired position easily. This movement is accomplished by the carriage drive transmission block which contains a semi-circular threaded hole, which mates with the rod during the actual operation of the carriage.

Figure 9 shows the film cassette being loaded with a 7 inch long, 3½ inch wide film. It will be noted that the cassette has a semi-circular profile. The semi-circular shape permits the cassette to travel along with the carriage previously described and not interfere with the entrance

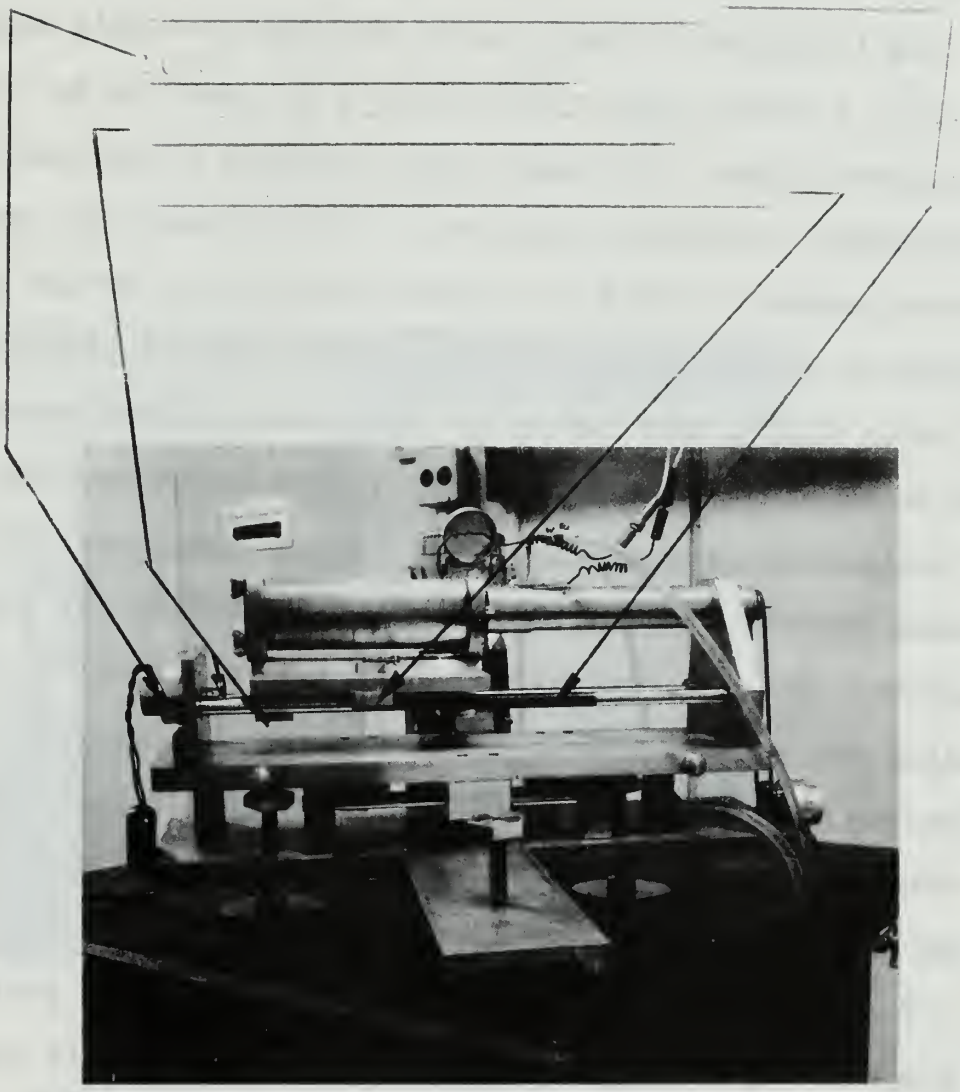
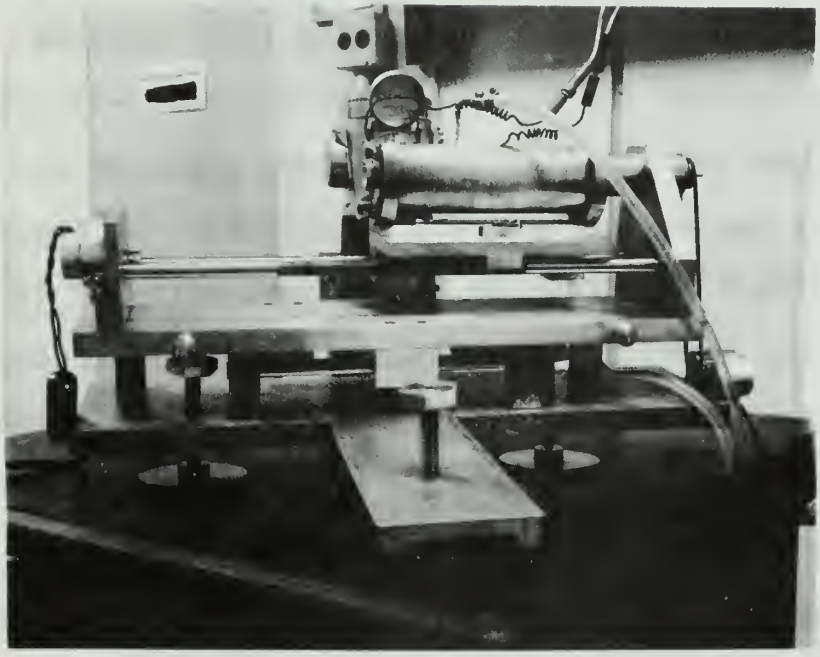


Fig. 9

collimator or wiring and cooling lines of the furnace. Cassette geometry and design basically follow the standard objectives as described by M. J. Buerger, Klug and Alexander, and A. Taylor in their books dealing with this subject. [5,9,14] A light tight film cassette is possible by using an aluminum sheet, .035 inch thick aluminum alloy used in lithography, previously employed in film holders for precession cameras. The film is held in a semi-circular configuration by this aluminum sheet which permits passage of the x-ray diffraction pattern. The aluminum does extract a cost in the longer exposure time required, but reduces the air scattering pattern that results from the added distance between entrance and exit collimators necessary for furnace location. Three screws secure a cover plate over the film loading end of the cassette. After exposure the film is extracted without difficulty from the cassette by tweezers. For short periods of operation the film length can be reduced accordingly.

The cassette is shown in its normal starting position on the carriage in Figure 8. The cassette assembly is free to slide between two guiding bars on the carriage to a desired location relative to the entrance collimator and then locked in place by means of a spring loaded handle. Figure 10 shows the unit as it looks at the completion of an experimental run. The time elasped for the position shown in Figure 10 relative to Figure 8 is on the order of 26 hours for specimens requiring a 30 minute exposure time and using a 1/8 inch slit focusing attachment.

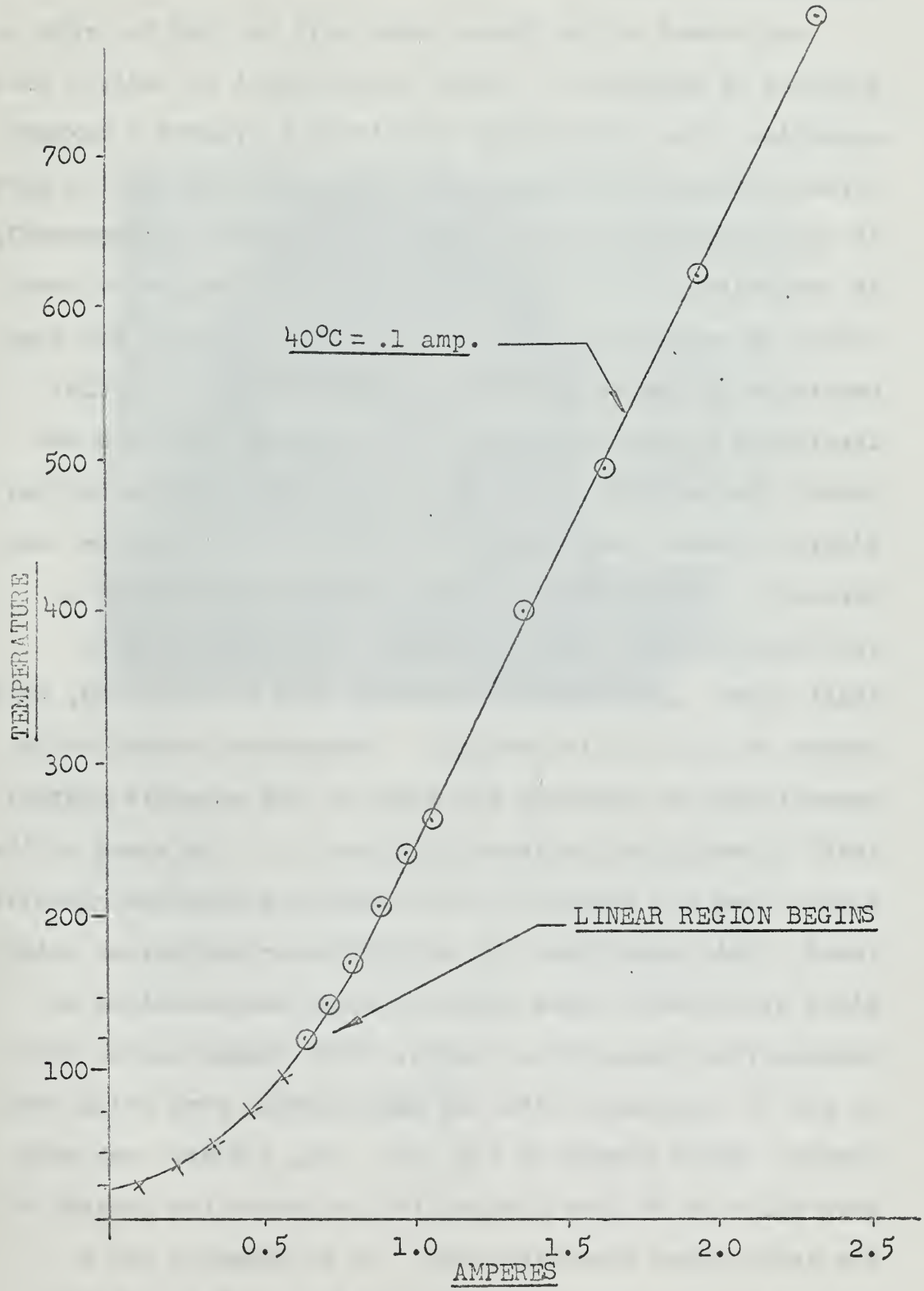


Apparatus in position following the completion of an exposure period.

Fig. 10

6. Furnace Operation and Programmed Heating.

The furnace, which was previously mentioned, is shown in Figure 5 in its operating position on the camera apparatus. The furnace is connected to a Kepco Inc. Mod ck 18 voltage/current regulated direct current power supply.[21] This power supply and furnace combination was used recently by Lt. McHugh in high temperature X-ray studies of a Cu-Be alloy. 26 The output current is constant during long extended operating periods and fluctuating line currents. Lt. McHugh calibrated the furnace using a Chromel-Alumel thermocouple and this calibration has been repeated and verified. The temperature-current relationship is shown in Figure 11. **Errors** specified by McHugh were expected to be no greater than 2.2°C for the Chromel-Alumel thermocouple. Heat loss due to the conduction of the thermocouple wires are minimal with regard to the small size of the furnace. These errors can be tolerated for the anticipated phase transformation surveys and the temperature-current calibration is quite adequate. However the temperature vs amperage for this thermocouple setup is not linear below 125°C. Therefore smaller temperature increments were used to define this region of the curve, as experimental work in this temperature range was expected. A more precise method of calibration, using materials with known phase transformation temperatures and described previously in the high temperature X-ray technology section of this paper, was used to verify this portion of the curve during the



FURNACE - CALIBRATION CHART

Fig. 11

experimental phase.

One aspect of the Kepco power unit is that the rate of increase of amperage is linear with respect to control knob operation. Ten revolutions of this knob produce a maximum current output of 3.0 amps, and each revolution of the knob is the equivalent of an increase of 0.3 amps. Consequently, as determined from the amperage-temperature curve, a revolution is equal to a rise in temperature of 120°C for temperatures in the range of 125 to 800°C. The versatile fractional speed synchronous timing motors again are employed for increasing and decreasing specimen temperature. Figure 12 shows the simple setup used to increase the temperature. A motor with a pulley and "O" ring making a frictional contact with a serrated knob on the shaft. Shaft speed, and resulting amperage rate is increased, by a factor of 1.5 in this exchange. Temperature reduction is accomplished by coupling the motor to the amperage control shaft directly and is shown in Figure 13. The speed of the motors used are selected to be compatible with the cassette speed. This means that for each exposure period, as determined by cassette speed and slit size, there will be an accompanying temperature change. This change may be high or low in accordance with the experimental work being conducted. Motor speeds of 1/2, 1/3, 1/6, 1/8 Rev. per hour were selected to give flexibility and cover the demands of the anticipated investigations. As an example, for a cassette motor speed of 1/4 rpm, using a 1/8 inch slit

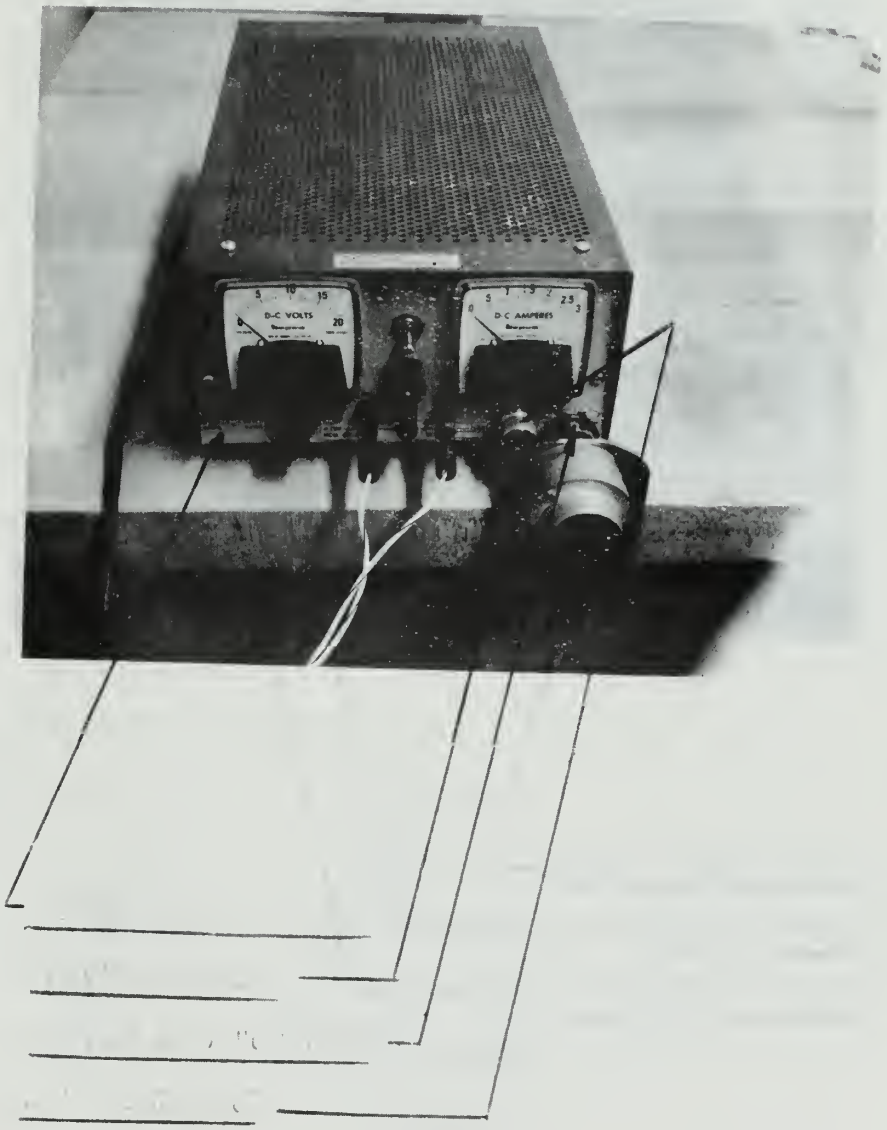


Fig. 12

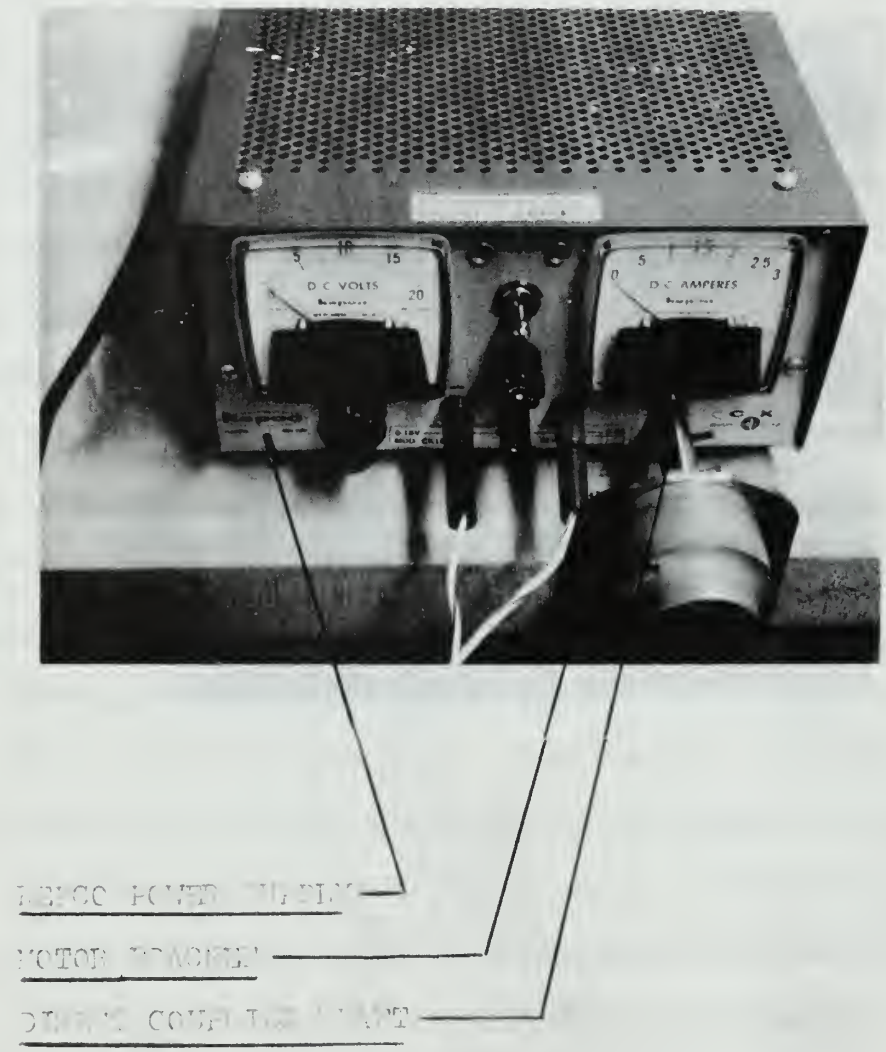


Fig. 13

focusing attachment, each equivalent exposure, as compared to single pattern cameras, would be 28 minutes long and encompass a 20°C temperature decrease in the direct coupling configuration using a 1/3 rev per hour power supply drive motor.

7. Experimental Investigation.

During the period of time when the camera was being designed, manufactured, assembled, and refined, the author devoted his spare time to improving his X-ray photographic techniques. Powder patterns of various specimens were taken using the Buerger High Temperature Camera shown in Figure 14.[4] Powder photographs of various specimens were taken at different temperatures, and would be used for comparison with the patterns obtained from the proposed camera. The initial objective, to build a high-temperature X-ray powder camera that would take a continuous series of X-ray diffraction powder patterns while the temperature of the specimen was increased or decreased, was kept in mind when different specimens were selected. Also the objective of recording a phase transformation on one sheet of film was used in specimen selection. Four specimens were selected to show that the designed camera and proposed concepts would satisfy these objectives, and show areas where the camera could be used. The specimen types selected were:

Alloy of unknown composition. The first specimen used was a piece of 1/8 inch diameter welding rod with unknown amounts of copper, zinc, and manganese. The rod was machined down to a thin (approx. 3/64 inch in diameter) solid rod type specimen. Powder particles of the welding rod were used to make thin collodion bonded specimens. Room temperature patterns of the collodion and rod type specimens were taken using the Buerger High-Temperature Camera (with

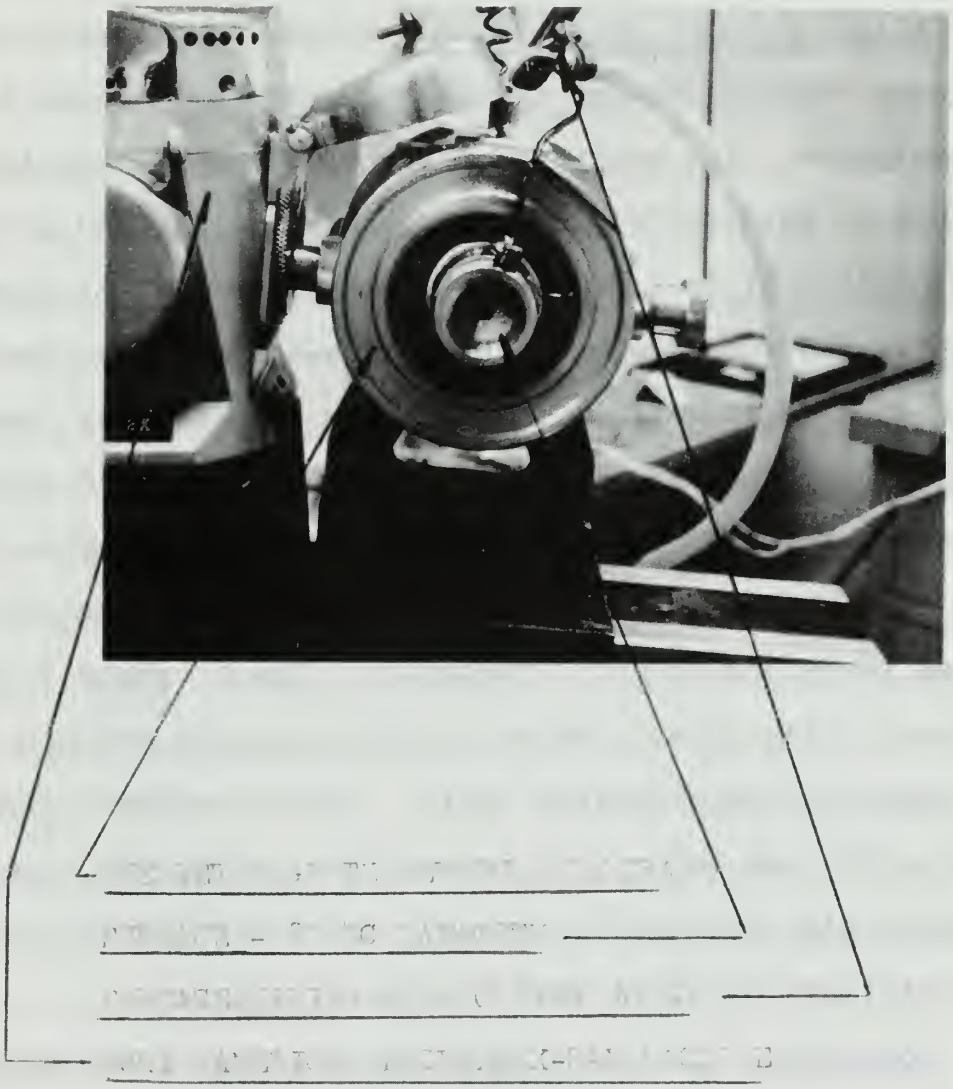


Fig. 14

and without the furnace attachment) and a conventional Debye-Scherrer Camera. For high temperature investigations, the Buerger Camera and rod type specimen were used to take photographs at various temperatures.

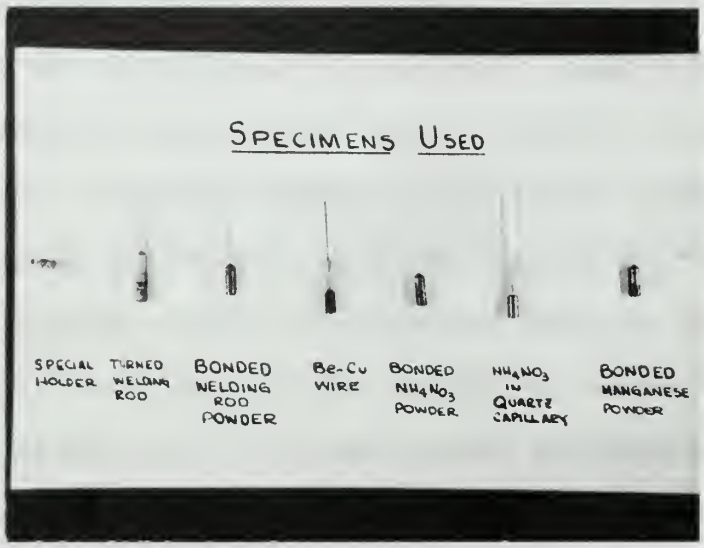
Alloy of known composition. A beryllium-copper alloy of known composition was used as the second specimen to be investigated. The analysis of the specimen as supplied by BERYLCO is Be-2.00,Co-0.22,Fe-0.14,Si-0.12,Al-0.03,Zn-0.03, Ag-0.01,Sn-0.01,Cr-0.01,Pb-0.00,Cu-balance (weight percent). The prepared specimen was a beryllium-copper wire previously drawn down to size by Lt. McHugh, who used similar specimens in his investigation of this alloy. The eutectoid transformation temperature for this material from the phase diagram presented in the Metals Handbook is 575°C where $\alpha+\beta$ phases transform to $\alpha+\gamma$ phases.[6] The α phase is Face-centered cubic, the β phase is Body-centered cubic, and the γ phase is Body-centered cubic. Powder patterns were taken above and below this temperature, using the single exposure high temperature camera, to be used for correlation with patterns obtained from the new type camera.

Non-Metal. The third specimen used was ammonium nitrate, which has known phase transformations near room temperature and gives a well defined powder pattern.[4,10] The specimen material was in the form of powder. The powder was further ground up and passed through a 325 mesh screen. This finely granulated powder was placed in thin walled quartz capillaries (.7mmI.D.) especially designed for powder

pattern work and distributed by UNIMEX-CAINE Corp., Chicago, Ill. These capillaries were sealed off to prevent oxidation of the specimen. Powder patterns at various temperatures were taken for later comparison. The following additional comments concerning the quartz capillaries are noted at this time. The quartz capillaries do not yield a diffraction pattern, but do reflect and absorb the X-rays to some degree resulting in longer exposure times. These capillaries are quite fragile, and must be handled with care. Their outside diameter is fairly uniform and special holders, as shown in Figure 15, were manufactured for their use. One end of the holder mounts in the eccentric device, used for centering, while the other end contains a hole, 1/2 inch deep for capillary placement.

Pure metal. Manganese metal, a well-known polymorphic with several transformation temperatures, the principal one of interest at 678°C, was used as the fourth specimen type. The metal available was very brittle and permitted only powder specimens. One specimen was prepared by sealing it off in a quartz capillary while another was mixed with a collodion to be used at room temperature for comparison with the capillary one. Powder patterns above and below 678°C were taken for later comparison.

Experiments with new camera. After receiving the newly designed camera from the machine shop, the assembly was mated to the North American Philips X-Ray unit. The specimens listed above for single pattern studies were



Capillary holder and prepared specimens used in development of a X-ray Camera that takes a continuous series of powder pattern photographs.

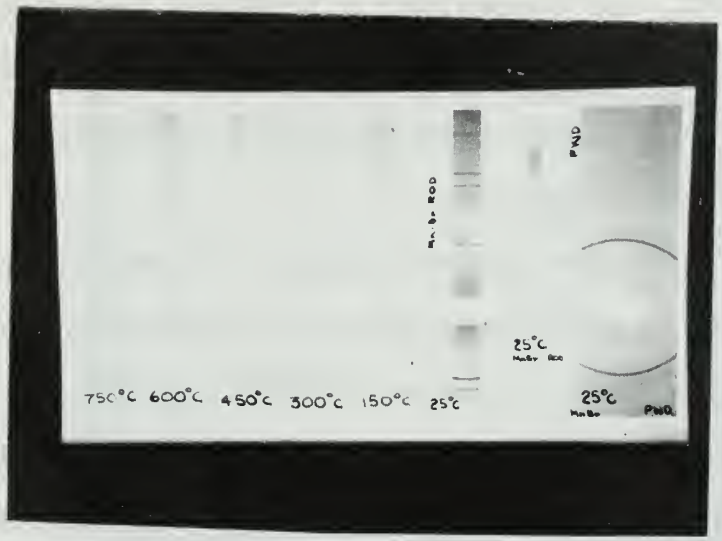
Fig. 15

used and continuous exposure powder patterns were obtained. The setup used for the camera is explained in the Description and Operation section of this paper. High and medium intensity copper radiation (maximum 50 KV and 26 ma) with a nickel filter was used for the welding rod, beryllium-copper alloy, and ammonium nitrate specimens. For the manganese specimen an iron tube (40 KV and 9 ma) was used with a manganese filter. Exposure times are too numerous to mention, but are labeled on the resultant powder pattern photographs. Various combinations of exposure times and heating rates were used to produce photographic patterns of a quality suitable for comparison and presentation. Also specimen preparations were varied to accomplish the same end result of producing diffraction powder patterns of defined quality. The specimens used are shown in Figure 15. The camera assembly's fractional speed synchronous timing motors and the Kepco power supply were electrically connected to the Philips X-Ray unit. By employing this electrical setup, it was possible to start the radiation, horizontal movement of the carriage, and programmed temperature changes at the same time. Completion of the exposure period and subsequent stopping of the above was accomplished by an electrical timer which is an integral part of the X-ray unit. This simple setup relieved the investigator completely for long periods of time with the exception of periodic equipment operation monitoring.

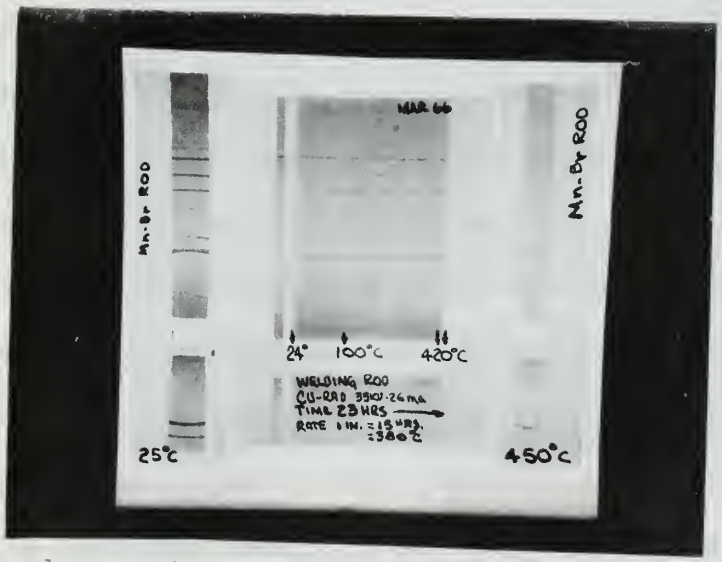
8. Results.

The best method of presenting the results of the experimental work conducted is pictorially. Figures 16 to 20 show the powder patterns taken with various cameras, different specimens, exposure times, and temperatures involved. It should be noted that the forward reflection is the only portion of the diffraction pattern recorded when using the new type camera. This is a result of cassette geometry and focusing attachment design. In addition, for comparison purposes, only the forward reflections obtained from the other cameras will be presented.

The powder patterns of the welding rod or alloy of unknown composition, Figure 16, reveals no phase transformation. The figure shows the same powder pattern at the different temperatures investigated for all types of cameras and types of specimens used. The patterns reveal some line broadening and lightening and is expected. The overall pattern obtained from the new camera resulted from a 1/16 inch slit focusing attachment, and a cassette speed of 1 inch every 14 hours. The power supply setup used for the temperature change is shown in Figure 12. The temperature increase was non-linear up to 125°C, or for the first 10 hours of the exposure period. Thereafter until completion the temperature increased at a linear rate of 24°C per hour. This implies that the film strip obtained from the new camera, as compared to single pattern cameras, contains the equivalent of 26 patterns with an exposure time of 52



Powder patterns of a Mn-Bronze welding rod specimen using conventional cameras and temperatures as indicated.



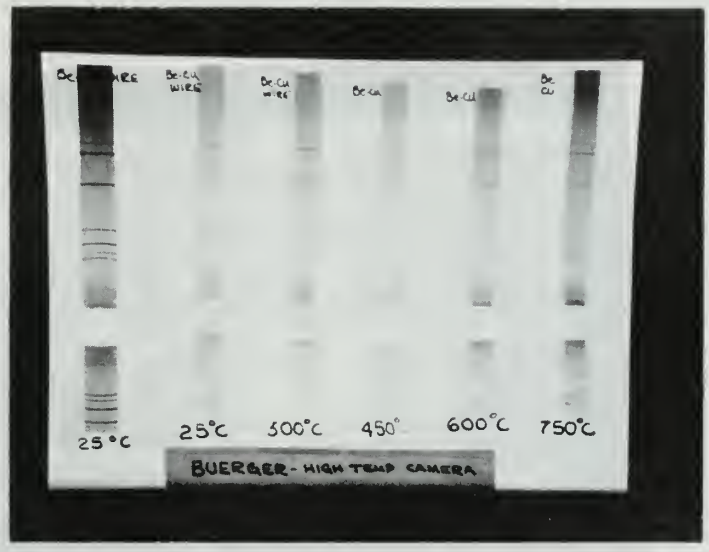
Powder patterns of the Mn-Bronze welding rod specimen using the Continuous Exposure and Buerger High-Temperature Cameras.

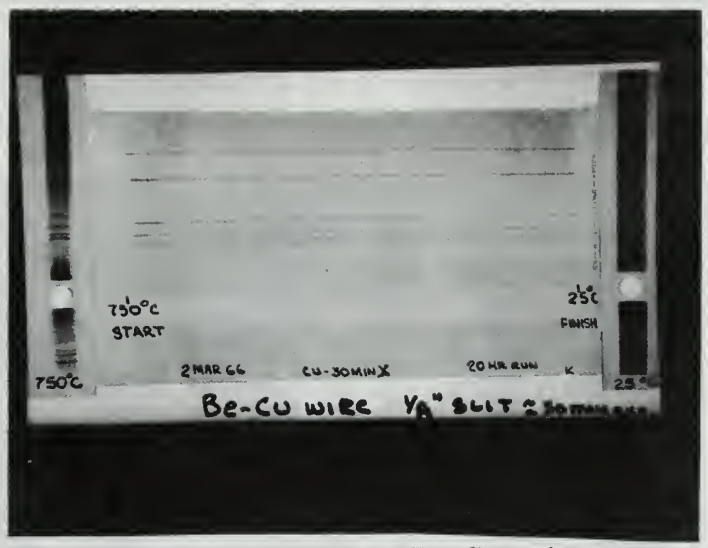
Fig. 16

minutes each. Each equivalent pattern, during the first 10 hours of the exposure period, covers an average temperature increment of 10°C, and thereafter a constant temperature increment of 21°C. There appears to be no slope to the diffraction pattern lines which should be indicative of a low rate of thermal expansion.

Figure 17 shows the powder patterns of the alloy of known composition, beryllium-copper, and reveals no pattern changes in the temperature range of 25°C to 750°C. The overall pattern obtained from the new camera resulted from a 1/8 inch slit focusing attachment, a cassette speed of 1 inch every 3 hours and 44 minutes, while the temperature was decreased using the power supply setup shown in Figure 13. The rate of temperature decrease was a constant 40°C per hour for the first 16 hours and an average temperature decrease of 24°C per hour until completion of the exposure period. The film contains the equivalent of 43 single patterns with an exposure time of 28 minutes each covering an 18°C temperature increment. The lightness of the resulting powder pattern lines is due to shorter exposure times and a lower intensity Cu target than was used in the welding rod experiment. A later test with a setup similar to that used with the welding rod specimen produced a sharper and darker pattern.

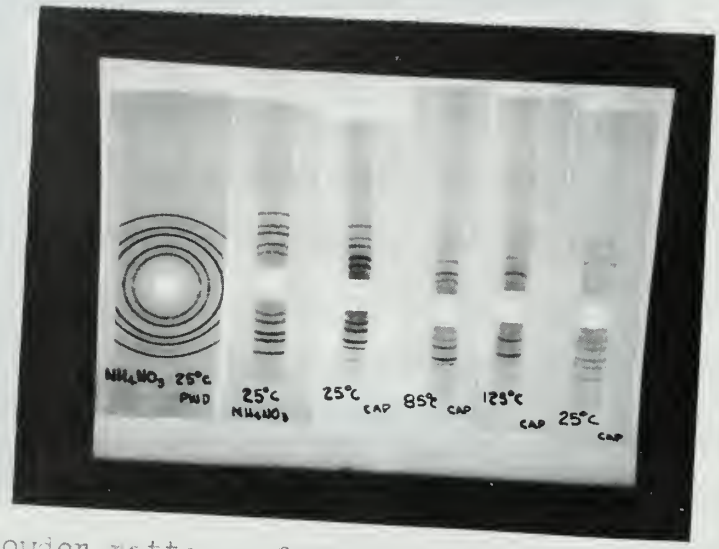
Figure 18 shows the patterns of the non-metal ammonium nitrate. The upper part of the figure compares powder patterns at various temperatures using conventional single



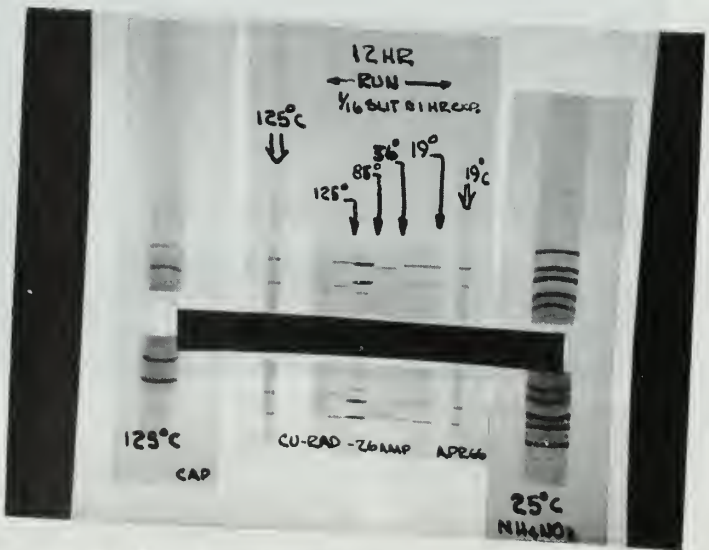


Powder patterns of the Be-Cu wire specimens using the Buerger and Continuous Exposure High-Temperature Cameras. Specimen temperatures and heating rates indicated.

Fig. 17



Powder pattern of ammonium nitrate using conventional cameras. Temperatures are indicated and phase transformations are evidenced by changes in the powder patterns.



Powder patterns of ammonium nitrate showing four different powder patterns on a single film strip as obtained with the Continuous Exposure Camera while specimen temperature is changed.

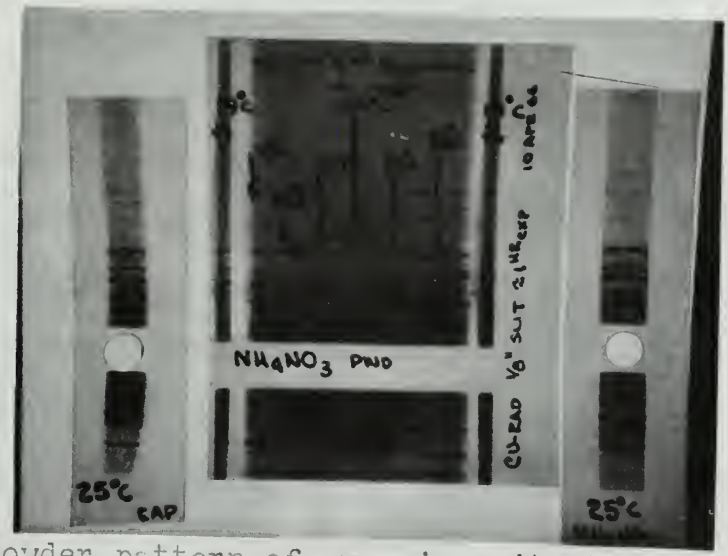
Fig. 18

pattern cameras. Differences in patterns are evident at 25°C and at 125°C. The continuous pattern obtained from the new camera, lower part of Figure 18, covers temperatures ranging from 19°C to 145°C and shows quite clearly pattern changes at 35°C, 85°C, and 125°C. These temperatures are approximate as the furnace calibration curve is non-linear at the low power supply setting involved. The temperatures were determined by measuring distance on the film strip and converting it to time from rate of advance information. Time was converted to amperage since the amperage rate was known. Then graphically from Figure 11, the temperatures relating to the determined amperages were obtained. Above 125°C the rate of temperature increase is constant (1 inch in horizontal travel is equal to 340°C), and direct methods can be employed. Broadening lines and lines of poor quality result from the difficulty in preparing a uniformly packed fine powder. The ammonium nitrate powder was water absorbent and caked together after it was screened before a good specimen could be prepared. Attempts at drying the powder using a kiln and desiccator produced no marked improvement in specimen quality. The continuous exposure pattern reveals quite well the different phase regions and transformation temperatures. It can be noted that at first glance the single exposure patterns at room temperature and at 85°C look strikingly alike, and indicate no phase transformation, but careful examination and measurement point out differences. This discrepancy is not noted

in the continuous exposure method and vividly points out an important advantage in using the new type camera.

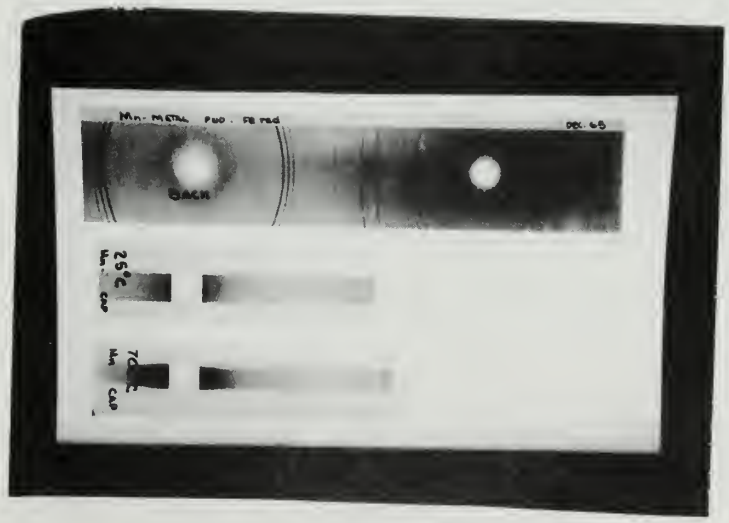
Figure 19 shows another ammonium nitrate diffraction pattern taken with the camera under discussion. Single exposure patterns included in the figure are for comparison purposes. The specimen was subjected to a two hour single exposure period at 19°C with no carriage movement, followed by a continuous carriage movement exposure period with a temperature increase to 110°C, then a temperature decrease, and finally a two hour single exposure period at 19°C. The 1/8 inch voids in between the patterns recorded result from a delibrate movement of the cassette on the carriage to accentuate the start and stop of the carriage movement exposure period. This feature along with the programmed increase and decrease in temperature on one film strip points up the versatility of the camera. The reversibility of the phase changes as indicated by the different patterns reveal that the material is allotropic and serves to show an additional camera feature.

Figure 20 shows the patterns resulting from the pure metal specimen of manganese powder. No phase transformation is evidenced from the patterns presented. Transmission patterns using single exposure cameras yield three weak lines. No pattern of any type was recorded using the new camera. Absorption by the aluminum film holder, capillary reflection, and relatively soft radiation from the iron target element are the factors contributing to no



Powder pattern of ammonium nitrate using the continuous exposure camera and illustrating the possible modes of operation. The increase, decrease temperature feature reveals the allotropic behavior of the specimen.

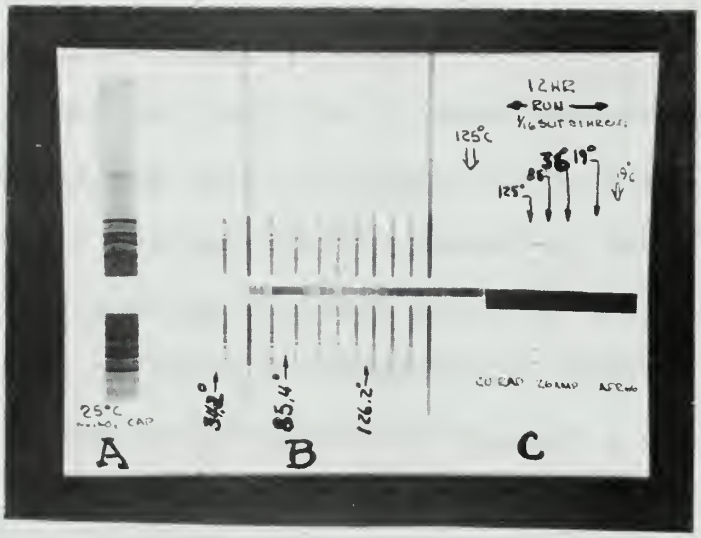
Fig. 19



Powder patterns of manganese using bonded specimens and capillary encased specimens at temperatures indicated.

Fig. 20

observed pattern. These factors can be overcome by refining the apparatus, specimen preparation, and radiation, and in so doing obtain manganese powder patterns with the new camera. Previous studies, by other workers, of manganese at elevated temperatures proved that the metal was too reactive to enclose in thin capillary tubes and too volatile to be photographed in vacuum. They overcame these difficulties by using a solid thin rod specimen and photographed in a pressure of several atmospheres of purified hydrogen. Time did not permit a similar setup by this writer as difficulty was encountered in producing a solid rod of the dimensions required from the manganese source on hand.



Lauda patterns obtained by the methods described.
A - Single exposure at one temperature.
B - Series of single exposures on one film strip at several temperatures.
C - Continuous exposures on one film strip covering entire temperature range.

Fig. 21

9. Summary and Conclusions.

(1.) From the resulting powder pattern photographs using the camera arrangement illustrated and described in this paper, the initial objectives of this thesis have been obtained. A camera was designed and developed to take continuous X-ray diffraction powder patterns of a specimen exposed to changes in temperature. Evidence of a phase transformation has been recorded and determination of the phase transformation temperature has been demonstrated and indicates one application for such an apparatus. The culmination of this work is illustrated in Figure 21 which compares powder patterns obtained by single exposure, series single exposure, and continuous exposure methods.

(2.) The furnace with its limited temperature range of 800°C and no provision for a controlled atmosphere are areas where work is needed to extend the versatility of the entire unit. Further studies would be useful to refine the cassette by replacing the aluminum with a different material to hold the film, and to investigate the possibility of using interchanging light tight film holders to serve as filters for the $K\beta$ radiation while using unfiltered characteristic radiation.

(3.) One area of research where this camera unit could be applied was demonstrated in this report and that is phase transformations. In that regard C. S. Barrett, about 10 years ago in work supported by ONR, discusses some theories of transformation in pure metals, where he

classifies transformations into two types.[8] In one type, martensite for example, the crystal alters its structure abruptly, a "snap" reaction, and in some cases an audible click is observed. In the other type transformation is characterized by progressive growth of the new phase through relatively slow migration of interphase boundaries. This process, a "sluggish" movement, is a nucleation and growth reaction. J. W. Christian in a recent paper discusses present theories of martensite.[27] The term "military" is used for the martensitic transformation or atomic rearrangement taking place in a disciplined fashion in contrast to the "civilian" phase change characterized by an independent random movement of atoms. These concepts and theories should be revealed by photographs obtained using the camera described.

(4.) Other areas where this camera and provisions for programmed temperature changes could be employed concern investigations of grain size, intermediate phases, precipitation hardening and lattice distortions.

(5.) Further refinements and additions to this camera could include extension to environmental variables beside temperature. Recording powder patterns when the specimen undergoes pressure differentials, changes in atmosphere and possibly conduct creep studies using thin rod type specimens and applying a load through a pulley and clamp arrangement.

10. Acknowledgements.

My advisor, N. W. Buerger, provided the seeds of thought for this endeavor and then continued to give advice, assistance, and understanding.

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The author is grateful to Mr. August B. Rasmussen, Model Maker for the Machine Shop at the U. S. Naval Postgraduate School, for his skillful craftsmanship in manufacturing an instrument of precision quality.

To Mr. Norman Walker, Head Model Maker of the Machine Facility at the U. S. Naval Postgraduate School, my appreciation for his cooperation and assistance during the manufacture of the camera components.

The Office of Naval Research provided the funds for the purchase of the "Kepco" power supply.

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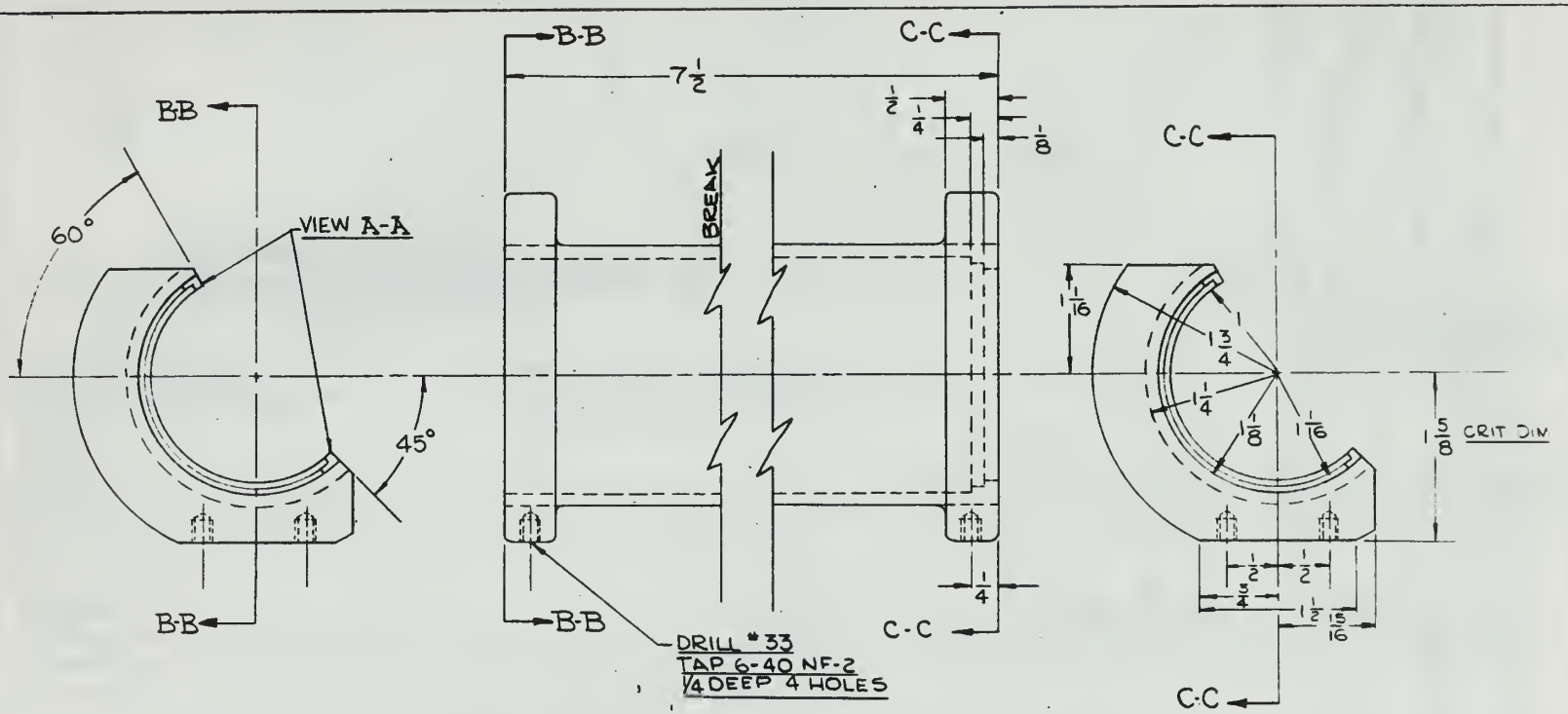
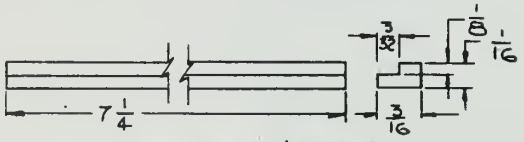
APPENDIX

This appendix contains working drawings for a X-ray Camera that takes a continuous series of powder pattern photographs while the specimen is subjected to programmed temperature changes.

APPENDIX CONTENTS





VIEW A-A
(SCALE 2TIMES)

"SWEAT" INTO PLACE
AS SHOWN ABOVE
2 PIECES

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PREP. KEIL		PART NO. MSX-3201N

A-2

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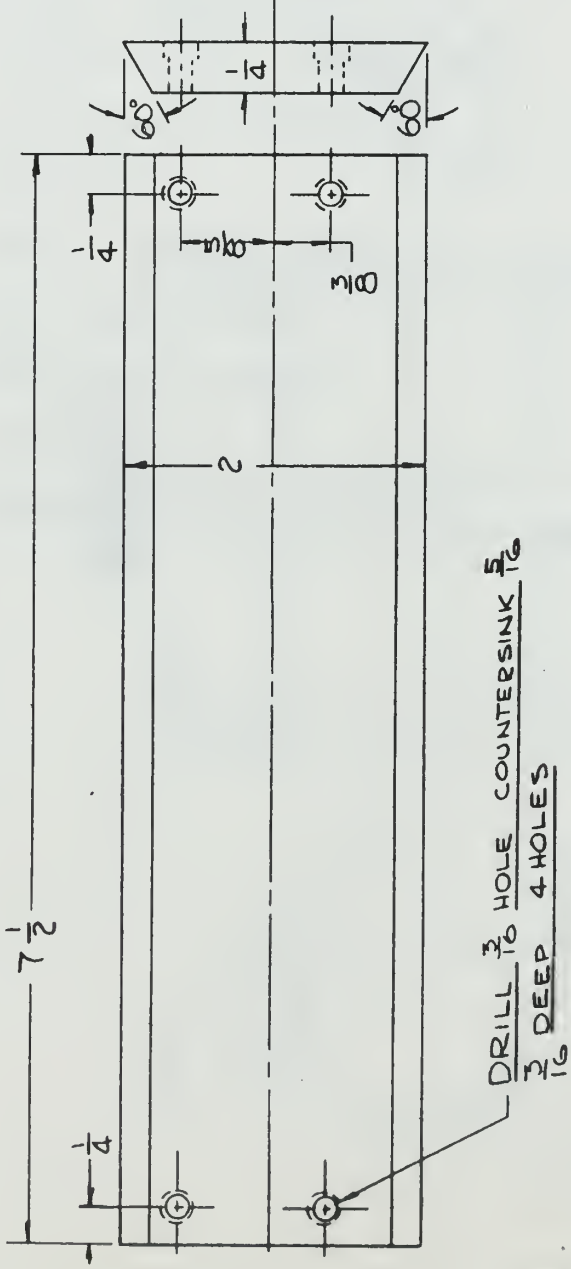

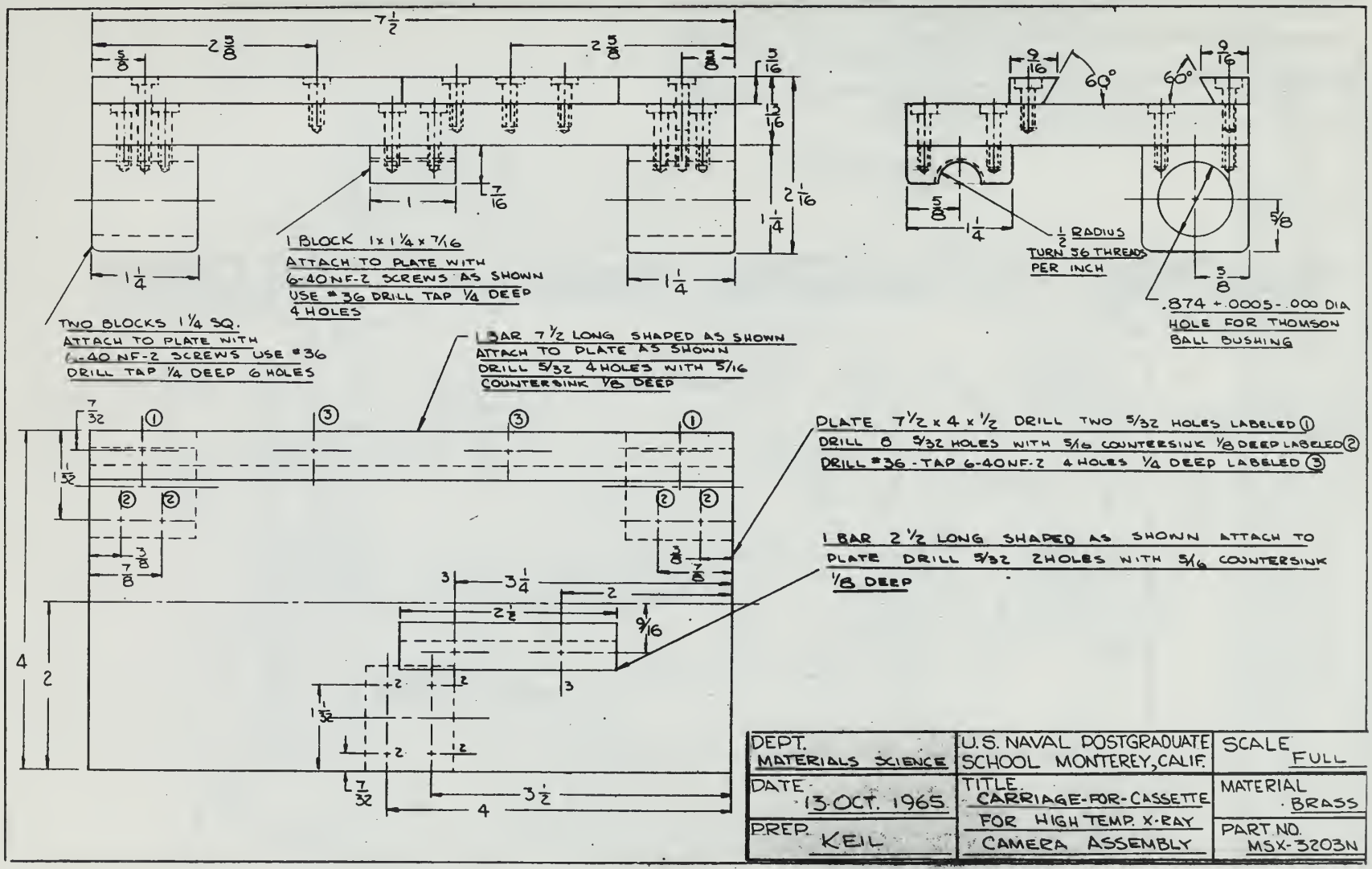

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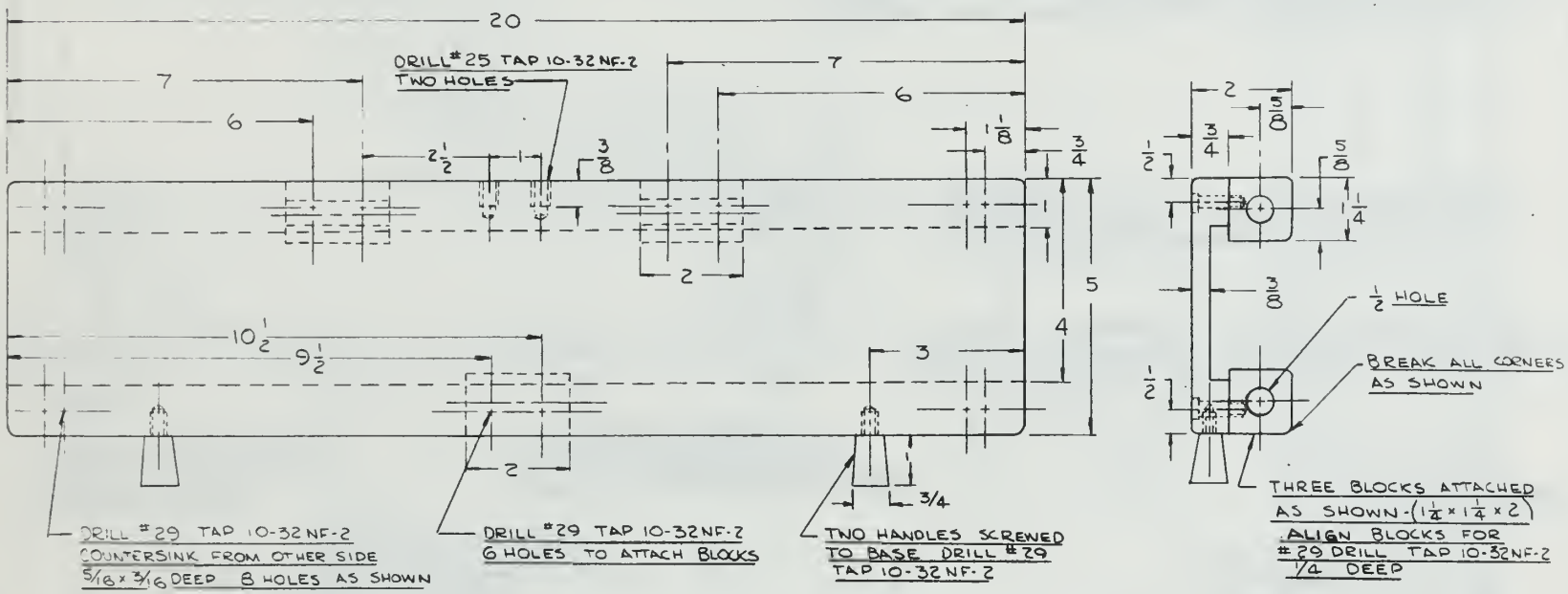

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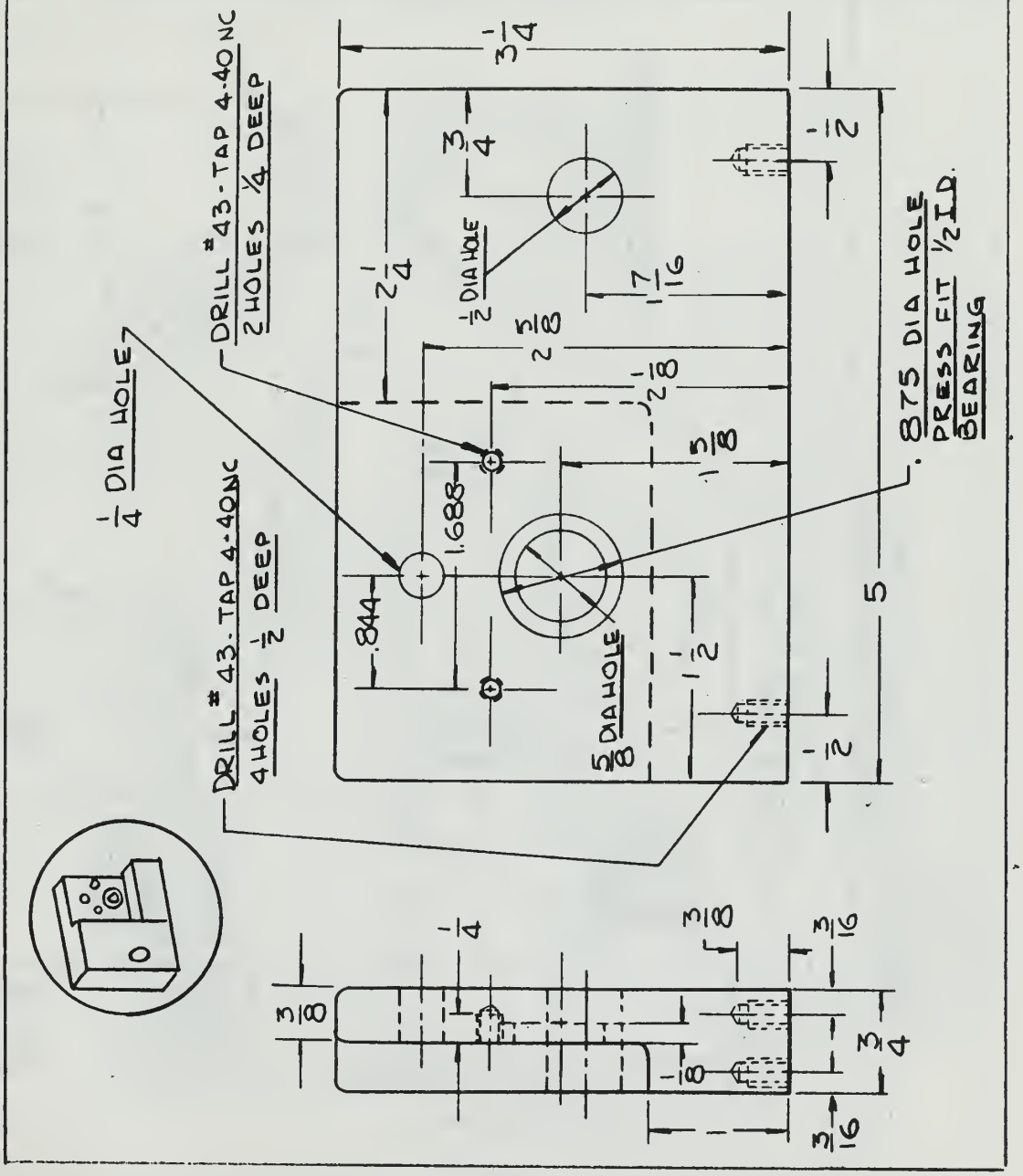

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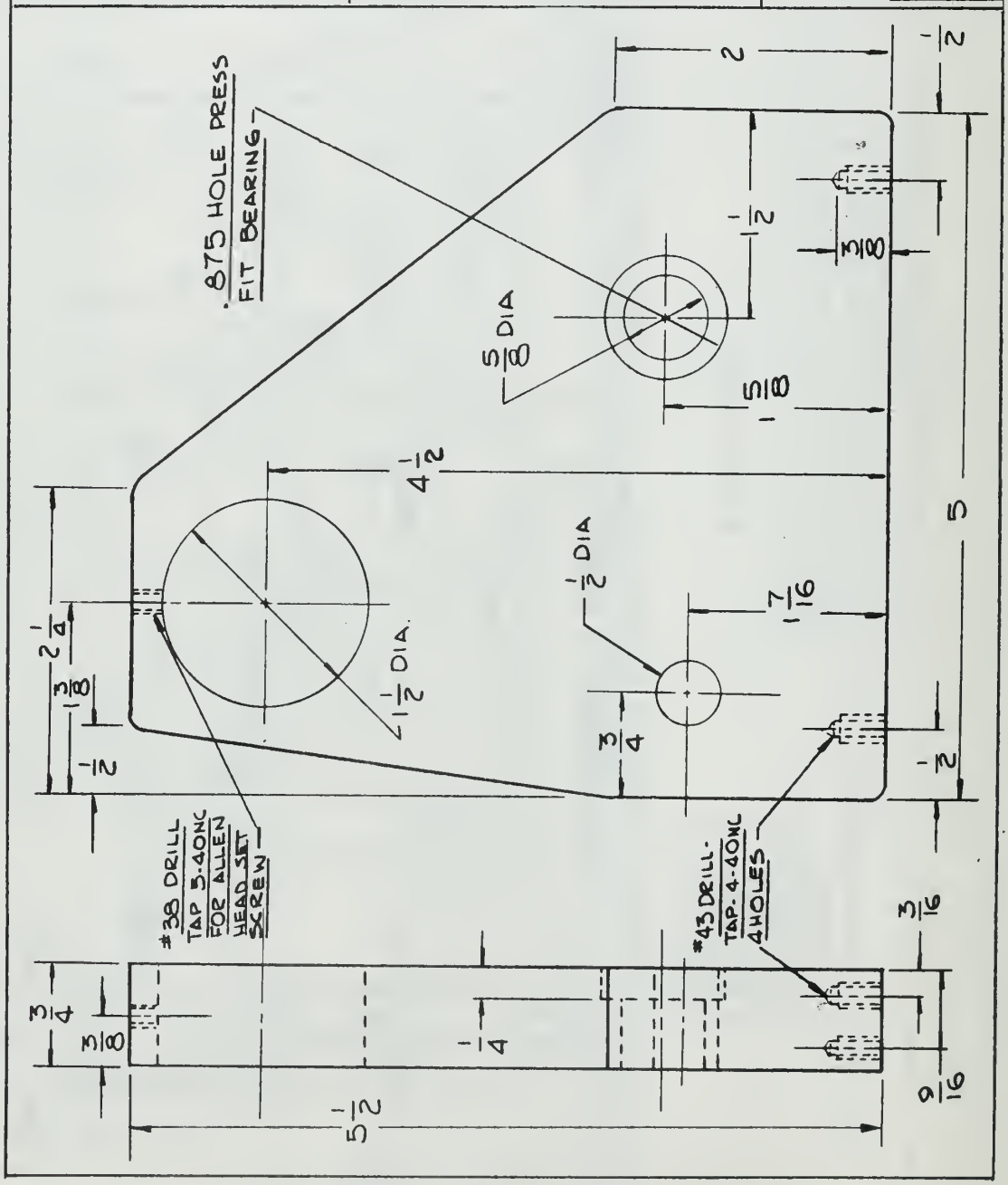

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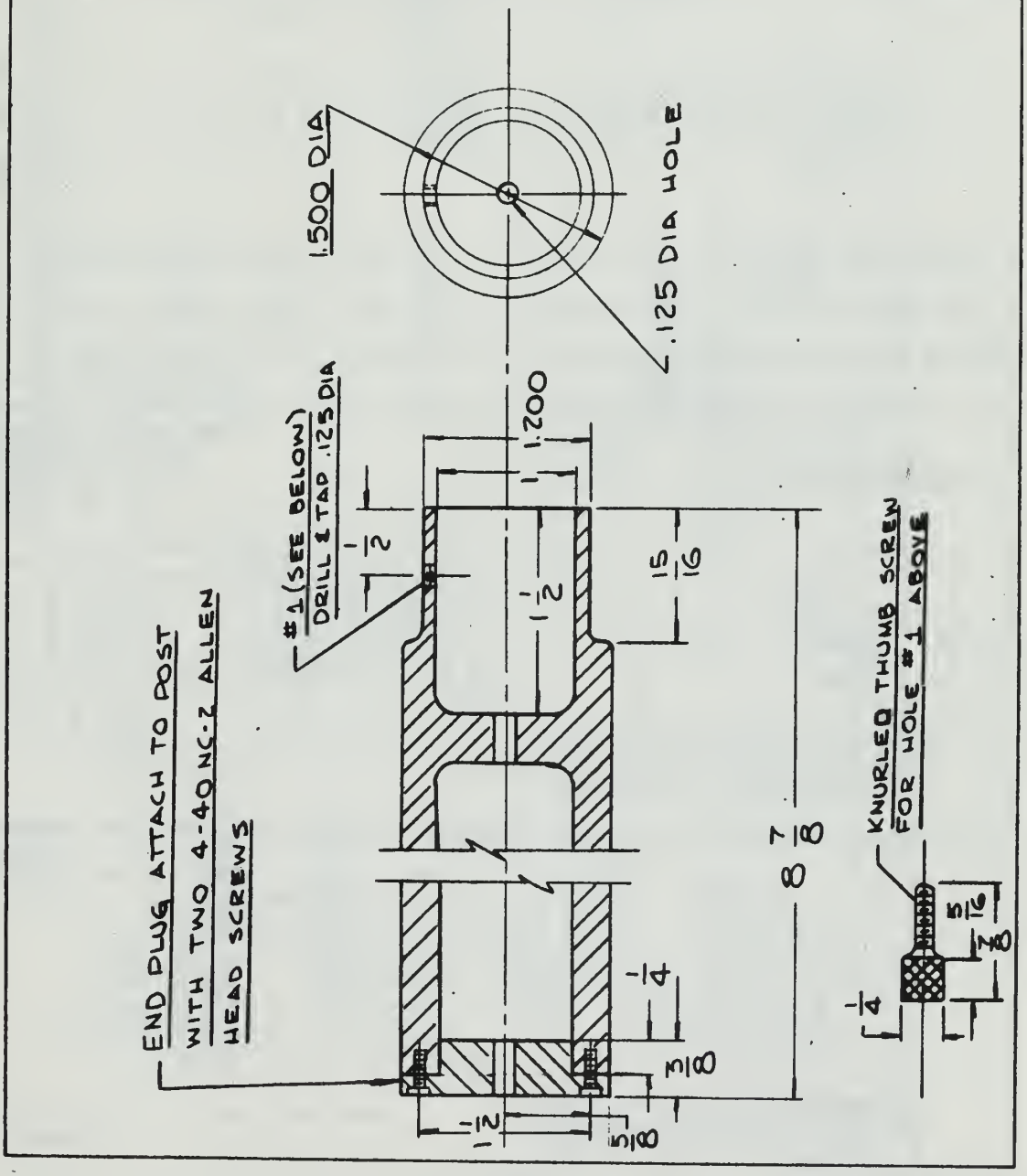
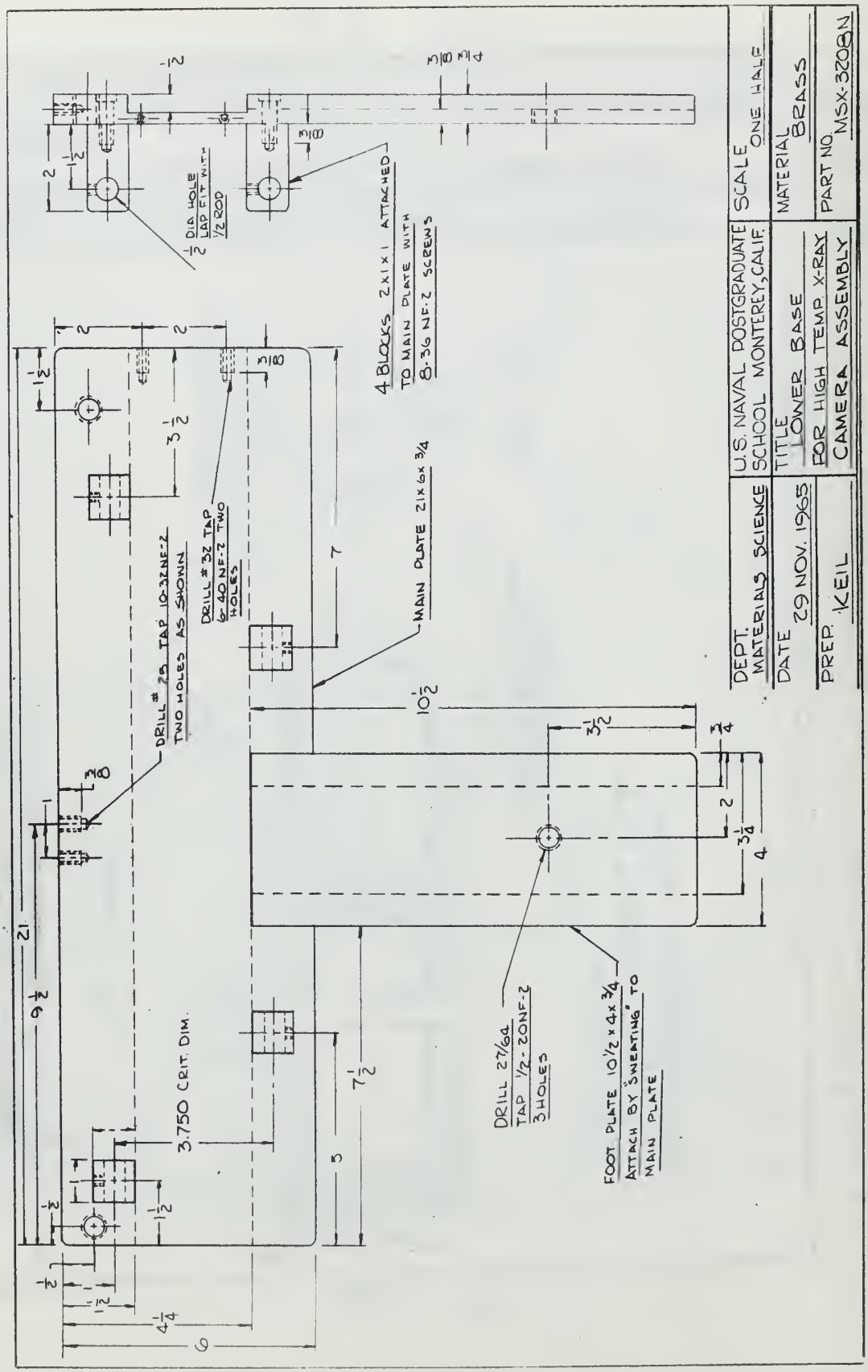
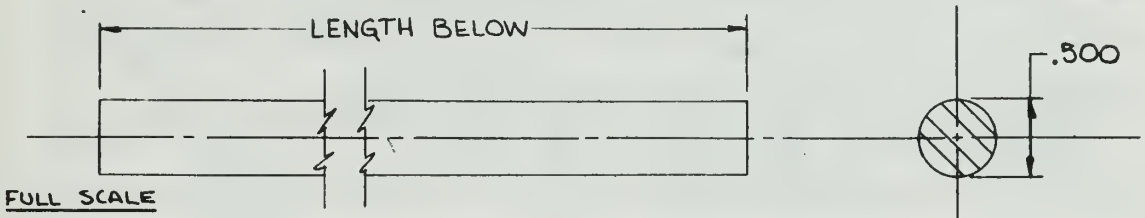



DEPT. MATERIALS SCIENCE
U.S. NAVAL POSTGRADUATE SCHOOL MONTEREY, CALIF.
SCALE ONE HALF
DATE 29 NOV. 1965
TITLE LOWER BASE FOR HIGH TEMP. X-RAY CAMERA ASSEMBLY
MATERIAL BRASS
PART NO. MSX-3208N
PREP. KEIL
4 BLOCKS 2x1x1 ATTACHED TO MAIN PLATE WITH 8-36 NF-2 SCREWS
MAIN PLATE 21x6x3/4
DRILL #25 TAP 10-32NF-2 TWO HOLES AS SHOWN
DRILL #32 TAP 6-40 NF-2 TWO HOLES
3.750 CRIT. DIM.
DRILL 27/64 TAP 1/2-20NF-2 3 HOLES
FOOT PLATE 10 1/2 x 4 x 3/4 ATTACH BY SWEATING TO MAIN PLATE
1/2 DIA HOLE LAP FIT WITH 1/2 ROD

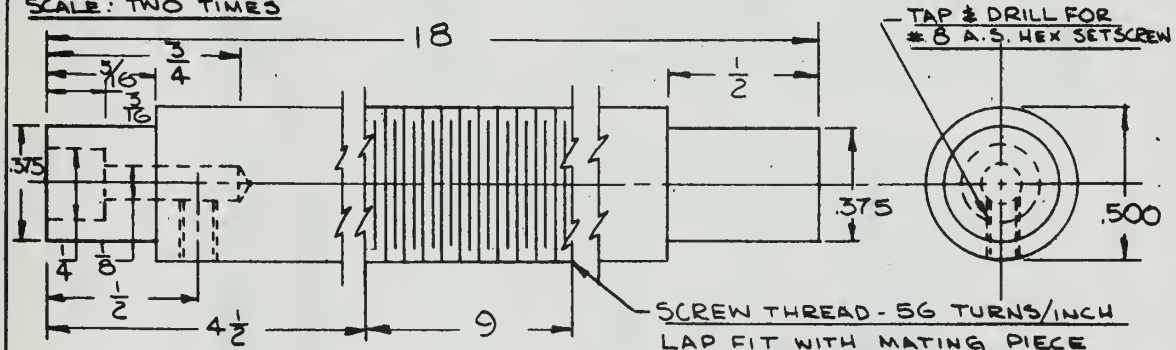
DEPT. MATERIALS SCIENCE	U.S. NAVAL POSTGRADUATE SCHOOL MONTEREY, CALIF.	SCALE FULL
DATE 29 NOV. 1965	TITLE GUIDE & DRIVE RODS FOR HIGH TEMP. X-RAY CAMERA ASSEMBLY	MATERIAL SPECIFIED
PREP. KEIL		PART NO. MSX-3209N



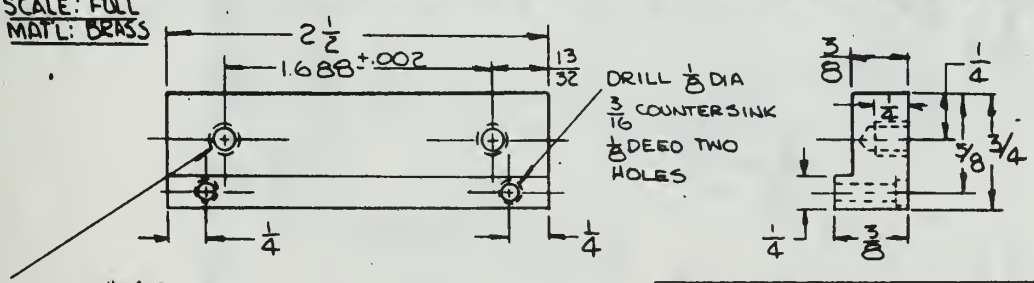
1. ONE STEEL ROD .500 DIA. 10" LONG LAP FIT WITH LOWER BASE
2. ONE STEEL ROD .500 DIA. 17" LONG LAP FIT WITH LOWER BASE
3. ONE STEEL ROD .500 DIA. 19" LONG FIT TO BEARINGS IN SLIDING SUPPORT BASE.

MATERIAL: STEEL ROD
SCALE: TWO TIMES

CARRIAGE DRIVE ROD



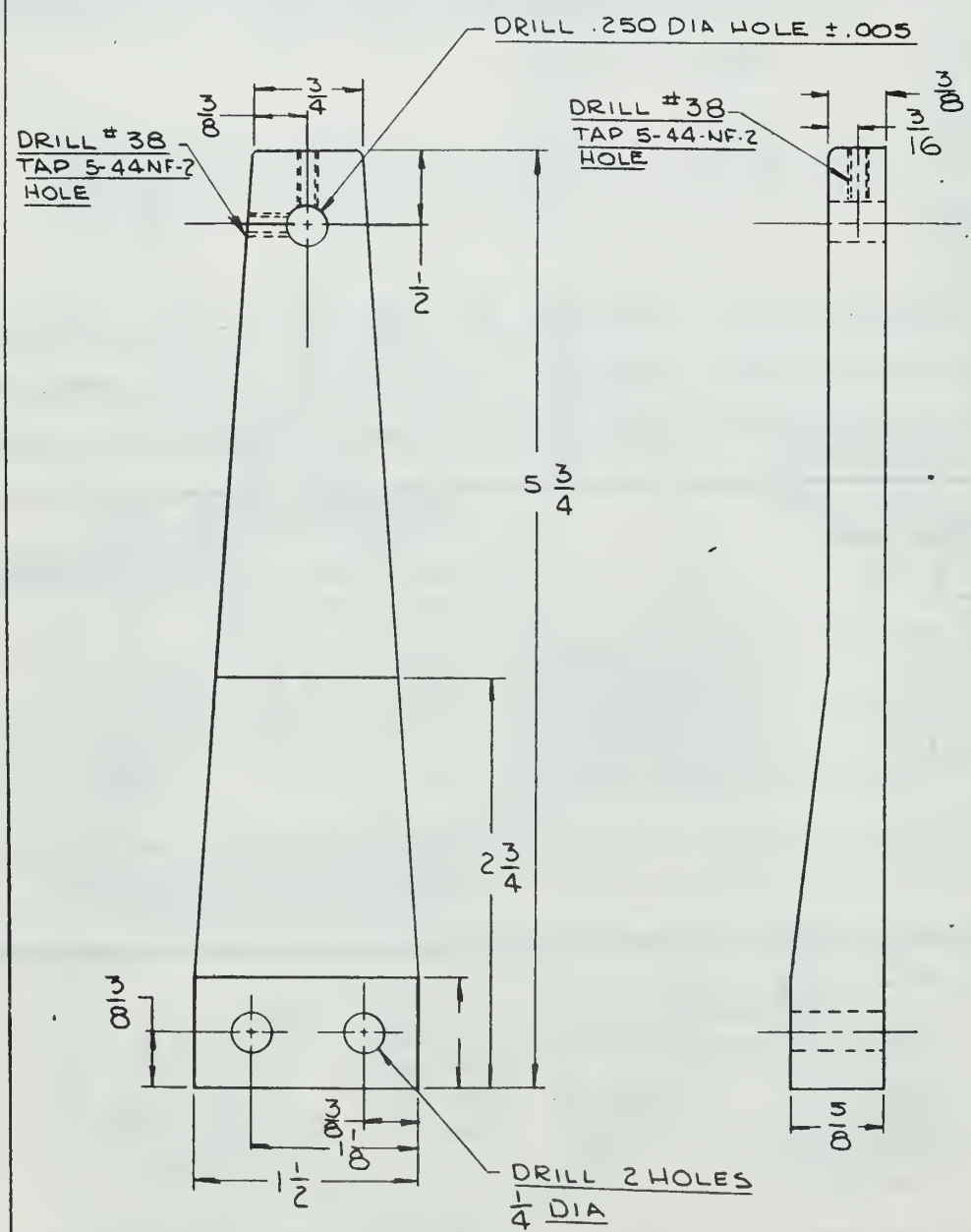
SCALE: FULL
MAT'L: BRASS



DRILL #43 - TAP 4-40 NC TWO HOLES AS SHOWN

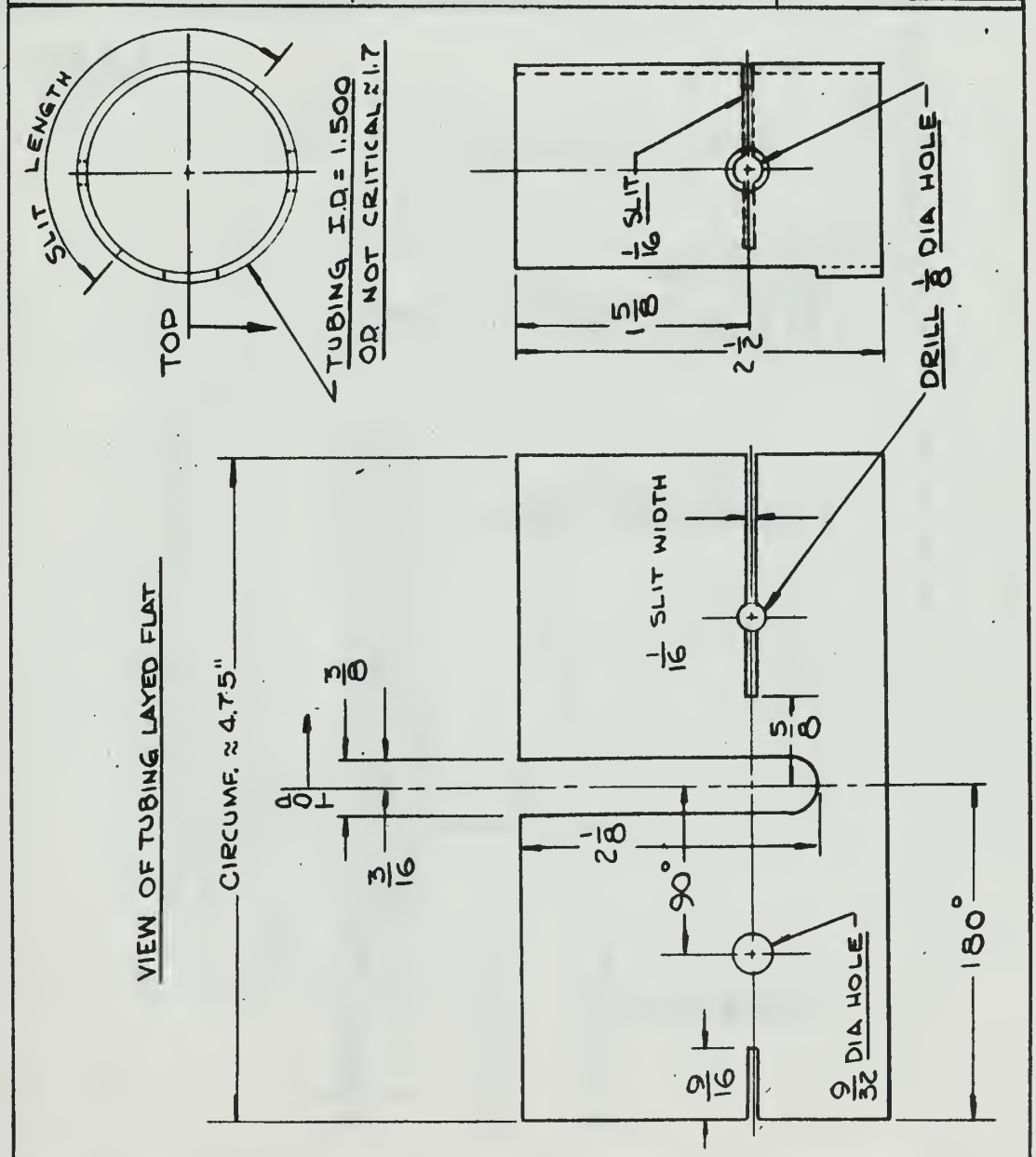
MOTOR BRACKET ATTACH TO LOWER BASE RIGHT END

DEPT. MATERIALS SCIENCE	U.S. NAVAL POSTGRADUATE SCHOOL MONTEREY, CALIF.	SCALE FULL
DATE 2 DEC. 1965	TITLE COLLIMATOR HOLDER FOR HIGH TEMP. X-RAY CAMERA ASSEMBLY	MATERIAL BRASS
PREP. KEIL		PART NO. MSX-3210N



A-11

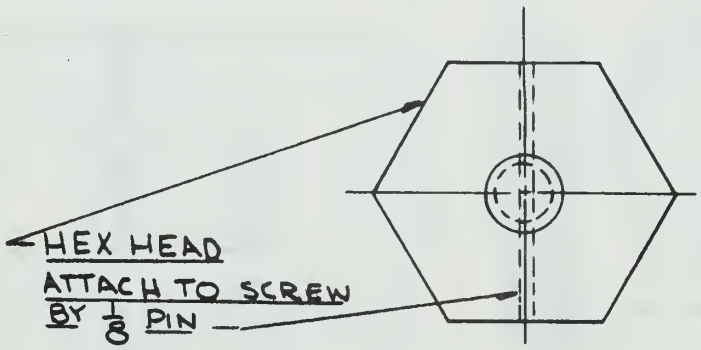
DEPT. MATERIALS SCIENCE	U.S. NAVAL POSTGRADUATE SCHOOL MONTEREY, CALIF.	SCALE FULL
DATE 9 DEC. 1965	TITLE FOCUSING ATTACHMENT FOR HIGH TEMP. X-RAY CAMERA ASSEMBLY	MATERIAL BRASS
PREP. KEIL		PART NO. MSX-3211N

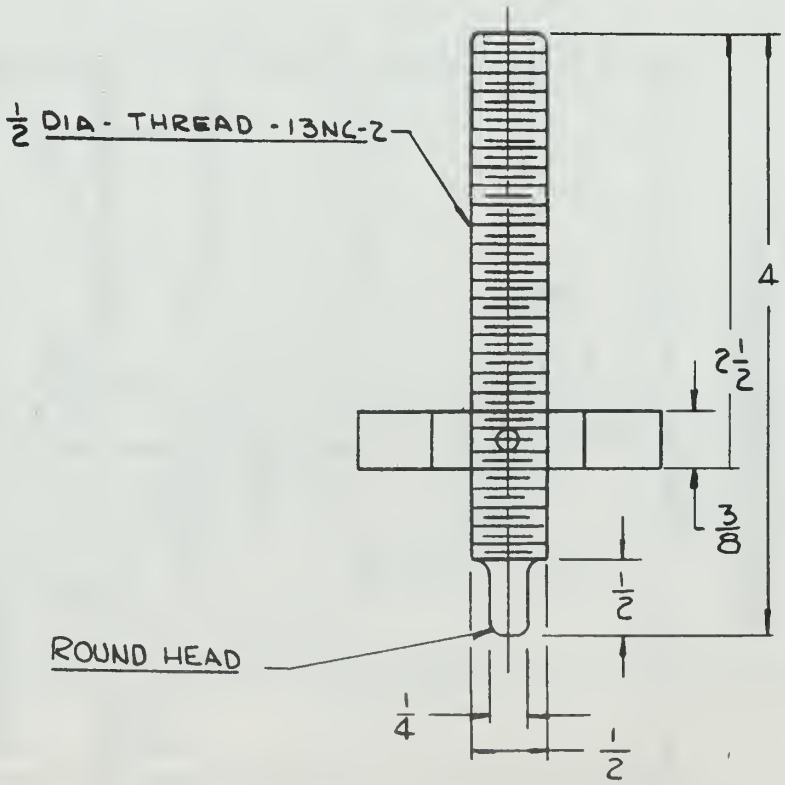


A-12

DEPT. MATERIALS SCIENCE	U.S. NAVAL POSTGRADUATE SCHOOL MONTEREY, CALIF.	SCALE FULL
DATE 9 DEC. 1965	TITLE LEVELING LEG FOR HIGH TEMP. X-RAY CAMERA ASSEMBLY	MATERIAL BRASS
PREP. KEIL		PART NO. MSX-3212N

3 REQ.





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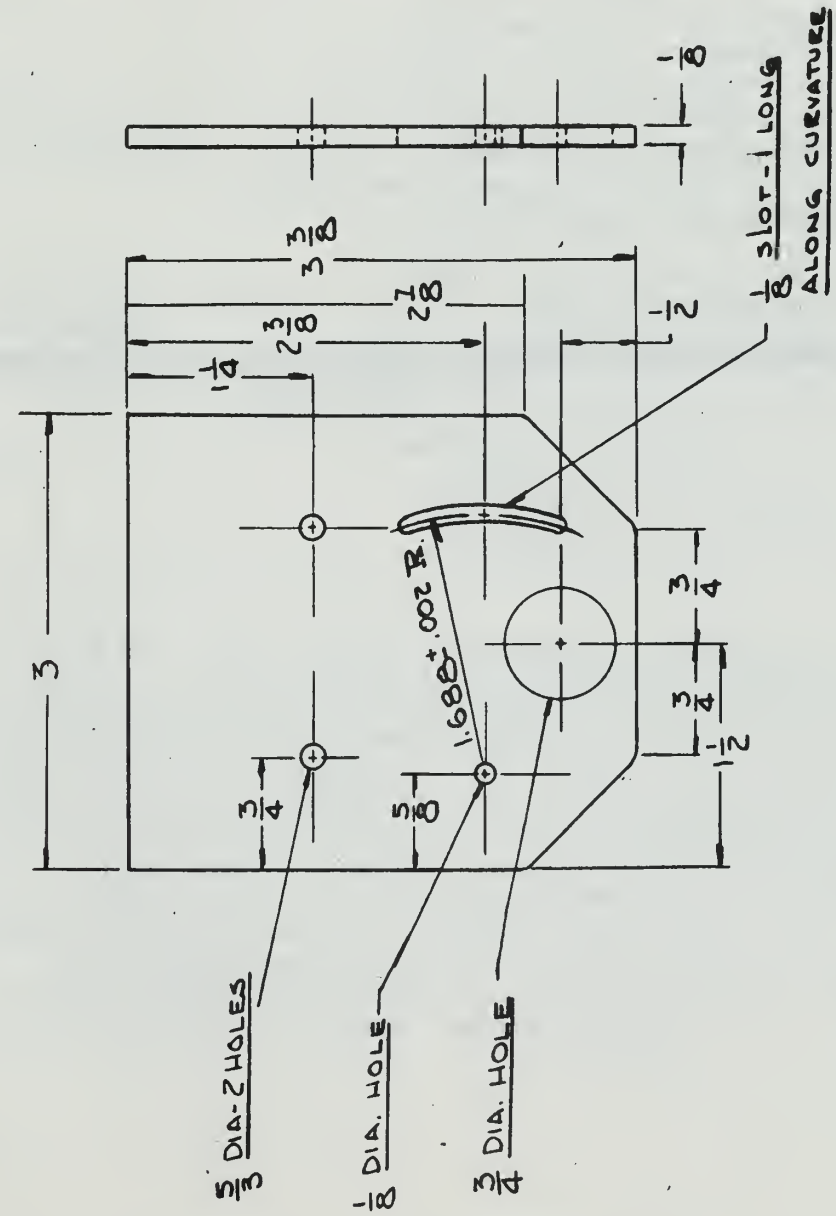
DATE 20 JAN. 1966	TITLE MOTOR BRACKET - USED WITH POWER SUPPLY IN HIGH TEMP X-RAY WORK	MATERIAL BRASS
PREP. KEIL		PART NO. MSX-3213N



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13. ABSTRACT

X-ray Analysis, using the Debye-Scherrer method, has been a laboratory tool for many years in studying the behavior of materials. The importance of performing investigations at high temperatures and changing environments led to the design and development of a X-ray Camera that extends the capabilities of basic powder pattern technology. A camera was designed to take a continuous sequence of X-ray powder photographs on a single strip of film while specimen temperature was changed. The design and development of this apparatus and its application in phase transformation studies is described and illustrated. The powder pattern photographs obtained with this apparatus provide desirable features and comparison presentation that can be applied to other investigations.

DD FORM 1 JAN 64 1473

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Apparatus for Continuous X-Ray Powder Patterns						
X-Ray Powder Camera for Elevated Temperatures						
High Temperature X-Ray Analysis						
Phase Transformation Temperature Powder Pattern Studies						

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